Influence of Moisture on Alkali Silica Reaction

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ABSTRACT

Influence of Moisture on Alkali Silica Reaction

Adeyemi Damilare, Adesina

Alkali silica reaction has been discovered over 75 years ago as a deleterious reaction in concrete, but the mechanism is not yet fully understood. Although there have been thousands of studies over the years, most of these studies have focused mostly on accessing aggregates that are susceptible alkali silica reaction so as to avoid it in new structures. In this study, focus is placed on understanding how moisture affects the progression of alkali silica reaction, as moisture cannot be eliminated in concrete compared to other prerequisites for alkali silica reaction in concrete. Understanding how various forms of moisture will affect the reaction will help to understand the reaction more and how to sustain structures already affected by ASR.

This study employs the use of relative humidity as a measure of moisture in concrete, four sensors were assessed and internal relative humidity in concrete samples was measured at different depths to see how the external conditions affect the internal moisture of the concrete. Also, the effects of the initial water added to concrete, i.e. the water to cement ratio, and external relative humidity was studied to see how they affect expansion due to ASR. The overall objective of this thesis is to observe how moisture in various ways affects progression of alkali silica reaction in terms of primarily expansions observed and visual inspection of samples under an optical microscope. It was observed that the external relative humidity has an effect on the internal moisture in concrete. Also, it was observed that the initial water to cement ratio has a slight increase on ASR as expansion of samples increase in water to cement ratio. It was also noted that fly ash and coatings

affect the internal relative humidity inside concrete.

ACKNOWLEDGEMENTS

I would like to show my sincere appreciation to my awesome supervisor; Dr. Nokken for believing in me and accepting to be my supervisor, working under you as brought a great positive development to me both in character and academically, your supervision allowed outstanding learning and growth.

Thanks to my friends who have contributed in one way or the other over the years to make this project as success, you guys rock! Finally, I dedicate this to my awesome mum.

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LIST OF ABBREVIATIONS

AAR	Alkali Aggregate Reaction
AASHTO	American Association of State Highway and Transportation Officials
ACR	Alkali Carbonate Reaction
ASR	Alkali Silica reaction
FA	Fly ash
HPC	High performance concrete
PC	Portland cement type I/II
RH	Relative Humidity
SCMs	Supplementary cementitious materials
W/C	Water – to - cement ratio

1 INTRODUCTION

1.1 GENERAL DESCRIPTION

For a concrete to be durable, it must exhibit a series of properties required for the particular environment to which the concrete will be exposed to during its service life. Since concrete is composed of different components such as cement, aggregate, water and admixtures, where necessary, the durability of these components has a future effect on the durability of the resulting concrete. Therefore, selecting materials to use in producing concrete for a specific application is a crucial step in avoiding durability problems as each material used for a specific concrete plays a key role for the concrete's durability.

One of the reactions that pose a threat to concrete's durability is alkali aggregate reaction; this is a type of reaction that occurs as a result of reaction between the components in concrete, mainly the aggregate and cement. Alkali aggregate reaction can be classified into two groups based on the reactants involved in the reaction: alkali carbonate reaction and alkali silica reaction. Alkali Silica Reaction (ASR) is a chemical reaction between alkali hydroxide in the concrete pore solution, generally derived from Portland cement and reactive forms of silica (SiO_2) found in certain aggregates (e.g., siliceous limestone, quartzite, greywacke, granitic-gneiss, and rhyolite). The reaction produces a hydrous alkali-silica gel, often referred to as ASR gel. Formation of the ASR gel alone does not cause cracking; however, when the gel absorbs water it shows significant potential to swell. The resulting expansion often results in pressure greater than what the concrete can withstand in tension, leading to concrete cracking. The best technique for the identification of

ASR is to examine a thin section of concrete using a petrographic microscope, also polished sections of concrete can be examined by scanning electron microscopy (SEM).

Alkali silica reaction can cause serious expansion and cracking in concrete, resulting in major structural problems and sometimes necessitating demolition. Since durability refers to the ability of concrete to resist deterioration from the environment or service in which it is placed, ASR is a serious threat to concrete because the resulting cracks created as a result of the reaction creates a pathway for other reactions such as corrosion or frost action, to occur in concrete.

Alkali-silica reaction (ASR) was discovered in the late 1930s and reported in the 1940s in California by T.E. Stanton, who first described it as a reaction between the cement and aggregate in concrete. It is now known that the reaction occurs due to the high pH of the concrete pore solution; this is a result of alkalis from the cement increasing the hydroxyl ion concentration to maintain charge balance within the pore solution.

The conditions required for ASR to occur are:

i) A sufficiently high alkali content of the cement (or alkali from other sources)

ii) A reactive aggregate, such as chert

iii) Water – ASR will not occur if there is no available water in the concrete, since alkali-silica gel formation requires water

Typical indicators of alkali-silica reactivity are map (random pattern) cracking and in advanced cases, closed joints and spalled concrete surfaces. Cracking usually appears in areas with a frequent

supply of moisture, such as close to piers, from the soil near retaining walls, near joints and free edges in pavements, or in piers or columns subject to wick action.

Because sufficient moisture is needed to promote destructive expansion, alkali-silica reactivity can be significantly reduced by keeping the concrete as dry as possible. The reactivity can be virtually stopped if the internal relative humidity of the concrete is kept low. In most cases, however, this condition is difficult to achieve and maintain.

Moisture does not only cause deterioration in only concrete, but almost on all building materials, e.g., steel, wood, etc. Moisture plays a major role in all forms of deterioration in most building material and cannot be totally eliminated being the most abundant fluid in nature and its ability to be in various forms such as snow, rain, ice, ground water, lake water, sea water and vapor made it easy to move through solids, therefore not only concrete is being affected by the deleterious effect of water. Water is known to have small molecules and also a universal solvent as it can dissolve more components than other solvents which made it easy for ions, gases and chemicals to dissolve in water, making it easier to attack solid materials and in this case concrete. Water's high heat of vaporization also contributes to its deteriorating tendency because it's able to exist in liquid state in porous materials rather than leaving the material dry by vaporizing.

Relative humidity is used in this thesis to measure the moisture content in concrete, it should however be noted that relative humidity is not the direct measure of moisture state in concrete. Based on this, moisture in concrete will be referred to as moisture or relative humidity in concrete frequently further in the thesis. Water is a part of concrete mixture from the time of mixing and

cannot be eliminated. Although water is required for cement hydration to bind fine and coarse aggregate; it also has the ability to cause physical and chemical degradation of concrete.

1.2 RESEARCH OBJECTIVES

The primary aim of this study is to understand the influence of moisture on alkali silica reaction in concrete. As concrete is a complex material and has an even greater complex microstructure, understanding how moisture exists in concrete is very critical to understand how it will affect the alkali silica reaction, because of this, a greater portion of this research is focused in finding ways to measure moisture in concrete by using different probes and sensors at different depths of the concrete sample. Therefore, the primary objective of understanding the influence of moisture on alkali silica reaction was further sub divided into the following objectives;

i) Test sensor suitability for measuring relative humidity inside concrete.

ii) Measuring of internal relative humidity in concrete samples with different properties and exposed to various external relative humidities.

iii) Investigating the effect of SCMs and coatings on the internal relative humidity inside concrete.

iv) Investigating the effect of external relative humidity on progression of ASR.

v) Investigating the effect of initial water content on the progression of ASR.

1.3 THESIS OUTLINE

The chapters of this thesis are outlined as follows;

The thesis starts with a general summary on concrete, its durability and a brief introduction to alkali silica reaction which is the focus of this thesis as chapter 1.

Chapter 2 covers the literature review which gives an in-depth examination into the durability of concrete in terms of alkali aggregate reaction with major focus on alkali silica reaction. Moisture measurement in concrete is also reviewed with previous studies on how moisture affects the reaction caused by the interaction between silica and alkali in concrete.

In chapter 3, the experimental procedures are fully explained including the materials, apparatus, and procedures employed for the four experiments. The experiments are grouped into two phases based on the material used for the samples, phase 1 is for experiments carried out in concrete samples while phase 2 is composed of the two experiments done on mortar samples. Chapter 4 show the results obtained also grouped into phase 1 and 2.

Chapter 5 comprises of all conclusions and recommendations from all the studies carried out and the thesis was finally concluded with the references of all sources consulted.

2 LITERATURE REVIEW

2.1 OVERVIEW

The most used building material in the world is concrete with billions of tons produced annually and used for different works in different sectors, ranging from transportation, health, education, recreation, and infrastructure.

Concrete is a composite material made of the different components; the components can be classified into two major parts which are the cement paste and aggregate. The paste part consists of cement and water while the aggregate part consists of both fine aggregate and coarse aggregate. In some cases, admixtures and supplementary cementitious materials can also be presented in concrete. These components also have effects on the fresh and hardened concrete properties.

Properties of concrete depend on its components and desired properties. Hence, it can be achieved by varying the amount of the following; water, cement, aggregate, chemical admixtures, waste materials and mineral admixtures as well as reinforcement. The amount of water used determines the water to cement ratio of the mixture design which directly affects the strength and workability of concrete. The greater the water for the same mixture design, the lower the strength of that particular concrete and the higher the workability. There are different types of cement, each with its unique properties. The mostly widely used is the General used (Type I) cement which is for general works. Other cement types can be used for conditions, i.e. mass concrete and/or improve durability of concrete such as sulfate resistance. The aggregates are mainly natural fine and coarse aggregate and they make up about 80% of the concrete. Also, some recycled products can be used as a replacement of aggregate.

Chemical admixtures are materials added to concrete to modify some fresh or hardened concrete properties. Chemical admixtures can be in powder or liquid form and have to be added in specific proportions to the mixture design to obtain the required properties. Some of chemical admixture types are; air retaining agents, plasticizers, accelerators, retarders, corrosion inhibitors, and pigments. Mineral admixtures are quite different from chemical admixtures, and as the name implies; they are inorganic materials and have pozzolanic or hydraulic properties. They are added to mixture design or blended with the cement during its production to obtain blended cements like ground granulated blast furnace slag, fly ash, silica fume and highly reactive metakaolin. These mineral admixtures are also referred to as supplementary cementitious materials (SCMs) when used as a replacement for cement in concrete.

2.2 CONCRETE DURABILITY

Durability of concrete is very critical from the selection of materials for concrete, to the mixing, placing, curing and maintenance of the concrete. The durability of concrete varies depending on the environment in which the concrete structure or element is being exposed and also the type of concrete properties desired. For example; the durability requirement of a rigid pavement in a cold region will vary considerably from a rigid pavement in an arid region; also, the durability requirement for a rigid pavement in a shipping port will be different from that in a residential area.

Causes of deterioration in concrete ranges from physical attack to chemical attack. According to Mehta and Gerwick (1982), physical attack can be grouped into two; 1) surface wear or loss of mass due to abrasion, erosion or cavitation and 2) cracking due to normal temperature and humidity

gradients, crystallization of salt in pores, structural loading, and exposure to temperature extremes such as freezing or fire. The chemical attack can be classified into three types which are; 1) cation exchange reactions between aggressive fluids and the cement paste, 2) hydrolysis of the cement paste by components of soft water and reaction attack, alkali – aggregate reaction and 3) corrosion of reinforcing steel in concrete. For this review, the focus will be only on one of the chemical forms of deterioration which is alkali aggregate reaction.

2.3 ALKALI AGGREGATE REACTION

As the name implies, alkali aggregate reaction is a reaction that occurs between alkali, which is mainly from cement, and an amorphous silica component present in the aggregate used for concrete. The reaction is simply depicted as shown in figure 2.1. This reaction was discovered as early as the 1930s in California in the United States; since then the effects have been seen in different countries all over the world.

Alkali aggregate reaction can be classified into two types, which are alkali silica reaction (ASR) and alkali carbonate reaction (ACR). These two types should not be confused with each other, though both have similar end result, but the process, duration and requirements for this reaction are totally different. Major focus will be placed on alkali silica reaction; therefore, only a short description and review of the alkali carbonate reaction will be made. However, it should be noted that ways of mitigating and preventing ASR does not work for ACR, also some of the test to detect the susceptibility of aggregates to ASR does not work for ACR.

Alkali aggregate reaction causes expansion in concrete which might lead to cracks resulting in a weak concrete and can result in other durability issues and even extensive damage to the structure leading to failure in the long run. The alkalis that exist in the pore solution are mainly from the Portland cement and can also be contributed by other sources like the environment, aggregates and admixtures.

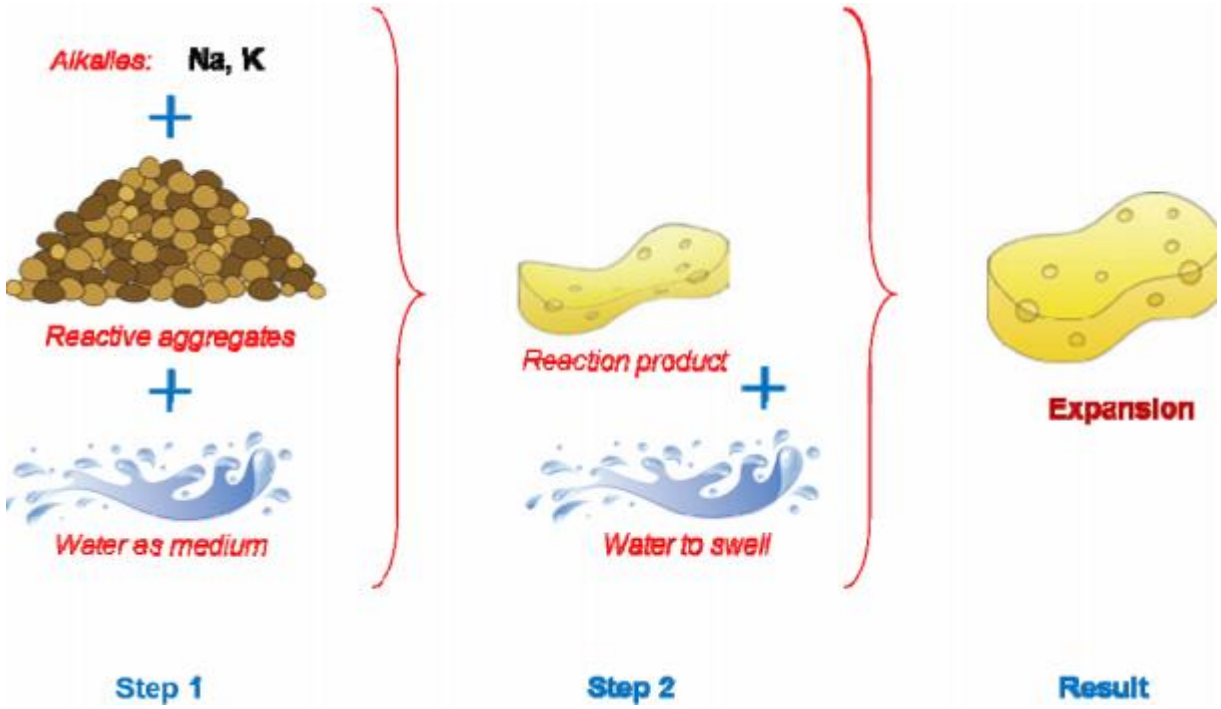


Figure 2.1 Steps involved in alkali aggregate reaction (Godart et al, 2013)

AAR does not only exist in North America; it has been reported in different countries all over the world like Denmark, China, Norway, United Kingdom, Australia, ranging from different structures such as bridges, rigid pavements, dams, buildings. Various studies (Dent et al,1981; Diamond 1983,1989,1992; Oberholster, 1986; Berube, 2000) over the years have helped to understand this reaction more and how it can be prevented in new structures.

2.4 ALKALI CARBONATE REACTION

ACR is a chemical reaction between the hydroxyl ion mostly from the cement and some specific carbonate rocks, especially dolomitic limestone and calcitic dolostone present in some aggregate. This alkali carbonate reaction mechanism can be represented in schematics shown in figure 2.2 results in the cracking as shown in figure 2.3. Dedolomitization of the aggregate which eventually leads to cracking and expansion in the concrete, this can be represented in the following reaction;

$$\underset{\text{Dolomite}}{CaMg(CO_3)_2} + 2\underset{\text{alkali}}{NaOH} \rightarrow \underset{\text{Brucite}}{Mg(OH)_2} + \underset{\text{Calcite}}{CaCO_3} + Na_2CO_3 \quad \text{……Eq. (2.1)}$$

There are few studies on alkali carbonate reaction and there has not been any agreement on its mechanism, the only agreement in this field has been pertaining to the physical and chemical composition of the aggregates involved. This reaction can be differentiated from alkali silica reaction in the following ways (AAR Fact book,2013)

- Supplementary cementing materials and pozzolans are not effective in preventing the reaction in new concrete works.
- Most tests for determine the reactivity of aggregate in ASR are not useful in detecting aggregate susceptible to ACR. (These test methods for ACR are presented in Table 2.1.)
- There are expansions at low alkali level compared to ASR where expansion only occurs at high alkali level.
- After expansion caused by ACR, no gel or little gel is found
- Damages from ACR can be observed in a short period of time compared to ASR.

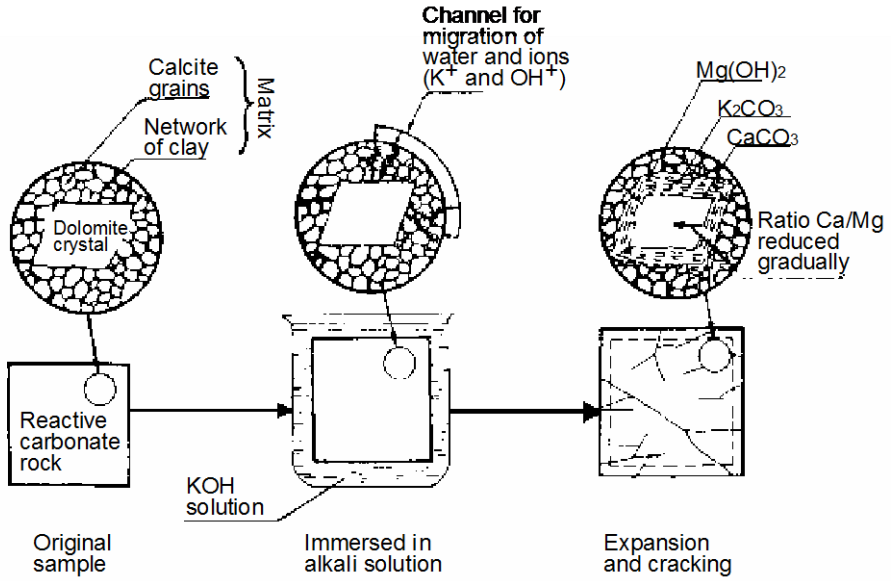


Figure 2.2 Schematics of alkali carbonate reaction mechanism (Farny et al, 2007)

Figure 2.3 Pavement affected by ACR (Farny et al, 2007)

The aggregates that are susceptible to alkali carbonate reaction have the following lithological properties (ACI 221 2000, Ozol 2006):

- Dolomite grains that are interlocked
- Carbonate fraction in the aggregate is between 40% to 60%
- Clay content in the range of 5% to 25%.

Alkali carbonate reaction can be mitigated by limiting the nominal maximum size of the aggregate (Swenson and Gillot, 1967), blending reactive aggregate with aggregates that are non-reactive and show good field record and using some inhibiting compounds such as lithium compounds (Gajda, 1996) but should be used in a good proportion as some of the lithium compounds has been found to increase expansion due to ACR (Wang et el, 1994)

Test name	Purpose	Type of test	Type of sample	Duration of test	Measurement	Criteria	Comments
Potential Alkali Reactivity of Carbonate Rocks for Concrete Aggregates (Rock Cylinder Method) (ASTM C 586)	To determine the expansive characteristics of carbonate rocks	Immersion of rock cylinders in alkaline solution at 23°C (73.4°F)	One rock cylinder, 35 mm long and 9 mm in diameter (1.38 in. long and 0.35 in. diameter)	Up to or over 1 year, but expansive tendencies are usually evident after 28 days	Length change	Not applicable	Securing representative sample may be difficult Can be a long test period lntended as supplement to other test procedures
Length Change of Concrete Due to Alkali-Carbonate Rock Reaction (ASTM C 1105)	To determine susceptibility of cement-aggregate combinations to expansive ACR	Concrete prisms stored at 23°C (73.4°F) and high relative humidity	6 concrete prisms 75x75x285mm (3X3X11¹/₂ in.) —or 100 mm (4 in.) square for 50 mm (2 in.) maximum size aggregate	1 year	Length change	Aggregate is most likely reactive if length change at 1 year (preferred measurement) is greater than or equal to 0.030% (0.025% at 6 months or 0.015% at 3 months)	Long test period Best used as a verifier of potential reactivity for a rock known to contain a potentially deleterious alkali-carbonate rock
Potential Expansivity of Aggregates (Procedure for Length Change Due to Alkali Aggregate Reaction in Concrete Prisms) (CSA A23.2-14A)	To evaluate the potential expansivity of both coarse and fine aggregates	Concrete prisms stored over water at 38°C (100.4°F)	3 prisms 75x75x285mm (3X3X11¹/₂ in.) and an additional (fourth) prism may be cast for petrographic examination	1 year	Length change	Aggregate is most likely reactive if length change is greater than 0.04% at 1 year	Long test period Use in conjunction with other information such as petrographic examination Similar to ASTM C 1293
Constituents of Natural Mineral Aggregates (ASTM C 294)	To give descriptive nomenclature for the more common or important natural minerals—a good starting point to predict behavior	Visual identification	Varies, but should be representative of entire source	Short duration—as long as it takes to visually examine the sample	Description of type and proportion of minerals in aggregate	Not applicable	These descriptions are used to characterize naturally occurring minerals that make up common aggregate sources
Petrographic Examination of Aggregates for Concrete (ASTM C 295)	To evaluate possible aggregate reactivity through petrographic examination	Visual and microscopic examination of prepared samples—sieve analysis, microscopy, scratch or acid tests	Varies with knowledge of quarry: cores 53 to 100 mm in diameter (2¹/₂ to 4 in.), 45 kg (100 lb) or 300 pieces, or 2 kg (4 lb)	Short duration—visual examination does not involve long test periods	Particle characteristics, like shape, size, texture, color, mineral composition, physical condition	Not applicable	Usually includes optical microscopy; may include XRD analysis, differential thermal analysis, or infrared spectoscopy—see C 294 for descriptive nomenclature
Petrographic Examination of Hardened Concrete (ASTM C 856)	To outline petrograhic examination procedures of hardened concrete—useful in determining condition or performance	Visual (unmagnified) and microscopic examination of prepared samples, acid etching	At least one core 150 mm diameter by 300 mm long (6 in. diameter by 12 in. long)	Short duration—includes preparation of samples and visual and microscopic examination	CaOH crystals are smaller than in undamaged concrete Are rims or partial rims depleted in CaOH? Pale color of rims may indicate gel	See measurement—this examination determines if ACR reactions have taken place and their effects upon the concrete Used in conjunction with other tests	Specimens can be examined with stereomicroscopes, polarizing microscopes, metallographic microscopes, and SEM

Table 2.1 Test method for aggregates susceptible to ACR (Farny et al, 2007)

2.5 ALKALI SILICA REACTION

Alkali silica reaction is a type of alkali aggregate reaction which occurs in concrete over time as a result of destructive reaction between chemical compounds present in the aggregate and cement used for concrete.

(a) (b)

(c) (d)

Figure 2.4 (a) Bridge column affected by ASR (www.wika.com), (b) Hydraulic Structure affected by ASR (www.jkmc.co.uk/) (c & d) Bridge components and rigid pavement affected by ASR (www.fhwa.dot.gov)

The reaction between these components leads to the formation of a product called silica gel which is hydroscopic, i.e. absorbs water, the silica gel expands in proportion to the available water present in the concrete thereby expanding leading to tensile stress in concrete. Some of the visual effects of this reaction that can be observed are surface pop outs, cracking, discoloration of the surface, expansion leading to displacement and deformation, gel exudations, etc.

This reaction has been discovered as far back as late 1930s by Stanton in California, who discovered that the alkali content of cement used to create mortar bars with some certain aggregates causes expansion in presence of moisture. He further discovered that cement with alkali level less than 0.60% does not cause expansion and the use of pozzolans can eliminate or reduce the expansion due to this reaction based on amount of replacement used. Based on discovery of alkali silica reaction by Stanton, several other studies were done on structures around United States and the reaction was found to be major cause of concrete deterioration in the United States.

Conclusively, three components are required before this reaction can occur to cause a deleterious effect:

i) A high alkali source mainly from the cement,
ii) A reactive aggregate, i.e. aggregates with sufficient quantity of silica, and
iii) Sufficient moisture.

2.5.1 COMPONENTS OF ALKALI SILICA REACTION

2.5.1.1 Sources of Alkali

Portland cement has been agreed to be the major source of alkali that gives the concrete's pore solution high alkali concentration and the quantity of alkali in Portland in cement can be determined. But in some other situations and environments, there are additional sources of alkali. These internal sources of alkali in a concrete can be summarized as follows

- Portland Cement
- Mixing water
- Aggregates
- Supplementary cementing materials such as slag
- Chemical admixtures

Alkali has to be present before this reaction can occur, elements such as lithium, potassium, sodium, rubidium (Rb) and caesium (Cs) are called the alkali metals and produce soluble hydroxide when mixed with water. Cement is the major contributor of alkali in concrete, it provides sodium and potassium. To obtain the alkali amount in concrete, Equation 2.2 can be used. It is not possible to simply add the sodium oxide (Na_2O) content and the potassium oxide (K_2O) content together because of the different molecular masses of the two oxides (Na_2O = 62.0 and K_2O = 94.2). The relationship between the alkali level in cement and alkalinity of the pore solution in concrete is shown in figure 2.5.

$$Na_2O\ equivalent(\%) = Na_2O(\%) + \left(\frac{62.0}{94.2}\right) K_2O(\%)$$
$$= Na_2O(\%) + 0.658\, K_2O(\%) \qquad \text{Eq. 2.2}$$

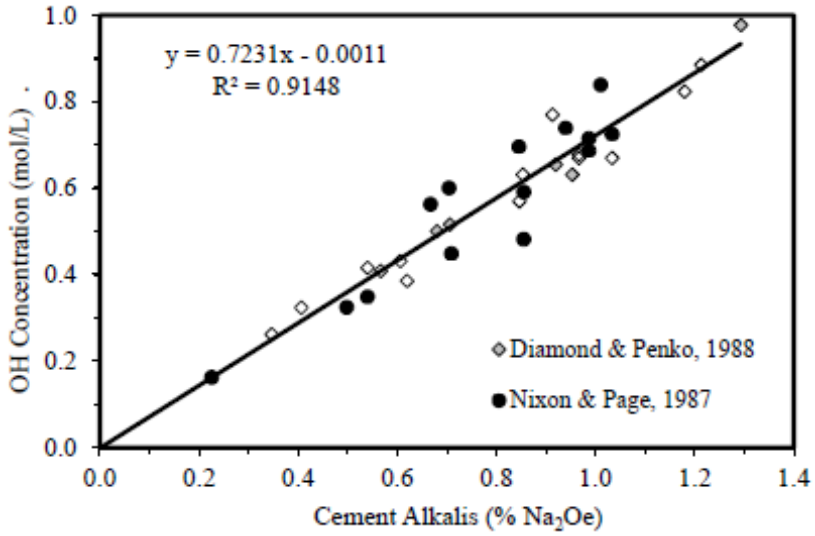


Figure 2.5 Relationship between alkali level in Portland cement and alkalinity of pore solution of concrete (AAR Fact book, 2013)

Though the presence of reactive aggregates in the concrete reduces the concentration of the alkalinity of the pore solution as a result of alkali silica reaction, the level of alkalinity still left is sufficient for the reaction. Figure 2.6 shows the effect of the presence of reactive aggregates on the pore solution alkalinity.

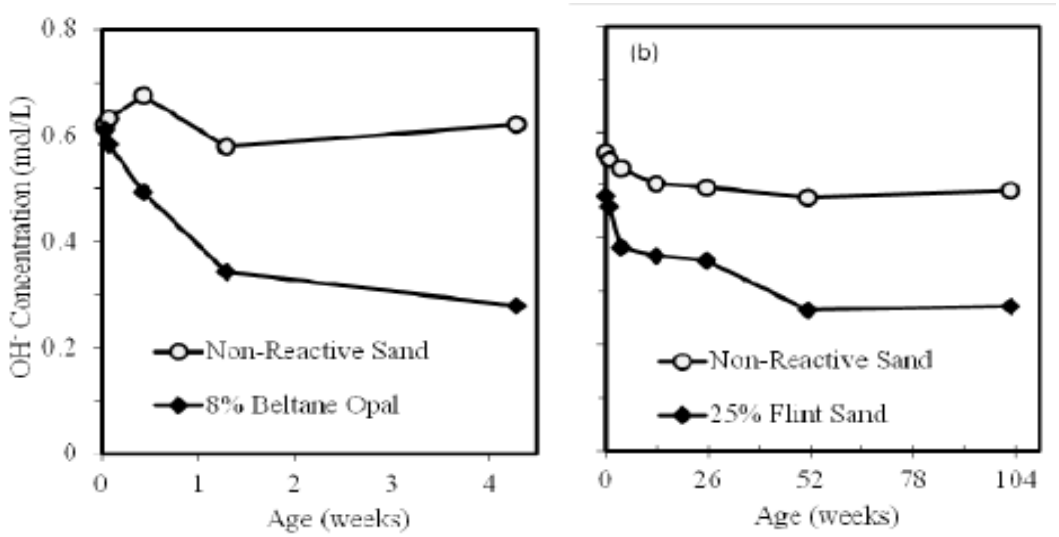


Figure 2.6. Pores solution alkalinity in presence of reactive aggregates

(a) Mortars and (b) Concrete (AAR Fact book, 2013)

Figure 2.6(a) shows the results observed by Diamond et al (1981) when reactive aggregate Beltane Opal was used at 8% replacement of aggregate, it was observed that the use of the reactive aggregate at the replacement level led to a reduction of the alkalinity of the pore solution with time, while non-reactive sand was used; there was just a little reduction in alkalinity of the pore solution which later increased to a steady level after time. Figure 2.6(b) shows a similar study by Thomas (1996) where he used flint sand which is a reactive aggregate at a replacement level of 25%. Similar results were obtained showing that reactive aggregates reduce the alkalinity of the pore solution. Diamond (1983) in another study stated that alkali concentration of 0.25M OH^- or higher is needed for alkali silica reaction to occur. The effect of alkali content of concrete on expansion of concrete prism stored at 38˚C over water was further shown by Diamond (2006) as shown in figure 2.7.

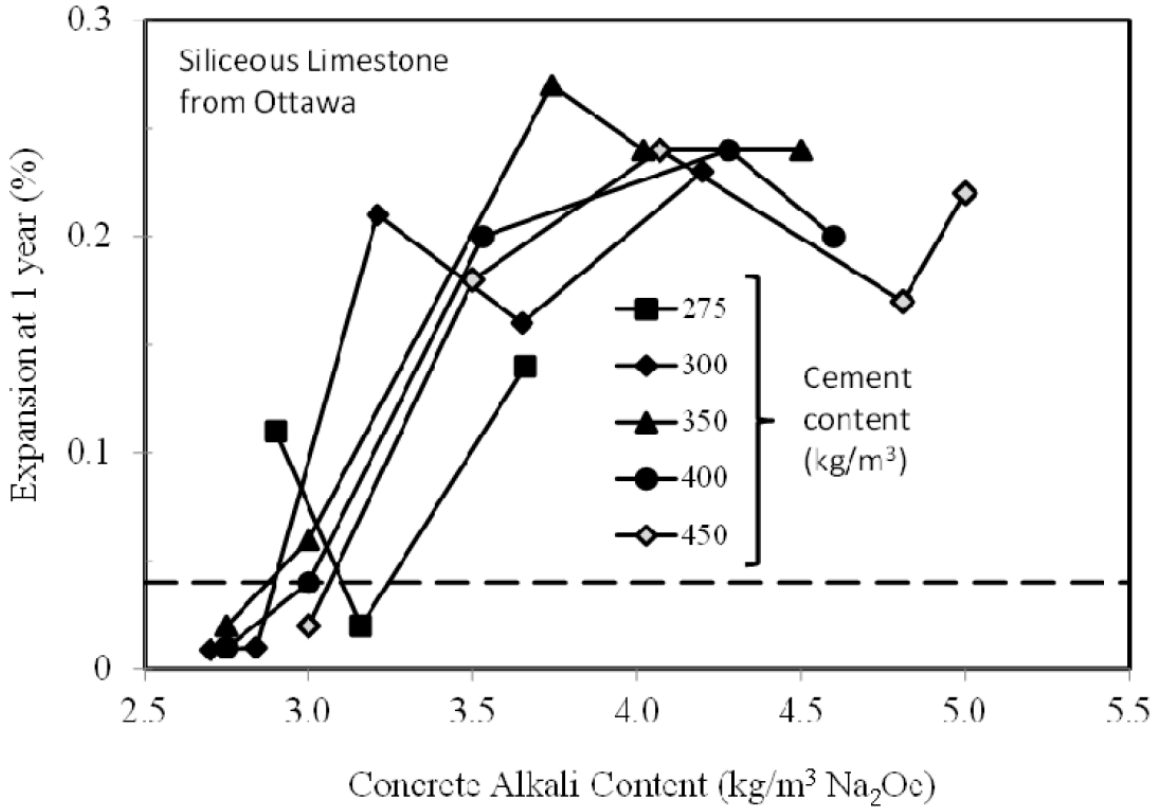


Figure 2.7 Expansion at 1 year based on different concrete alkali content (Diamond, 2006)

2.5.1.2 Aggregates

Different aggregate compositions will depend on location and stratigraphy from which they are obtained. This variation in composition leads to aggregates that are reactive and non-reactive in the context of alkali silica reaction. Aggregates can be classified based on several composition and properties.

Reactive substance (mineral)	Chemical composition	Physical character
Opal	$SiO_2 \cdot nH_2O$	Amorphous
Chalcedony	SiO_2	Microcrystalline to cryptocrystalline; commonly fibrous
Certain forms of quartz	SiO_2	Microcrystalline to cryptocrystalline; crystalline, but intensely fractured, strained, and/or inclusion-filled
Cristobalite	SiO_2	Crystalline
Tridymite	SiO_2	Crystalline
Rhyolitic,dacitic, latitic, or andesite glass or cryptocrystalline devitrification products	Siliceous with lesser proportions of Al_2O_3, Fe_2O_3, alkaline earths and alkalis	Glass or cryptocrystalline material as the matrix of volcanic rocks or fragments in tuffs
Synthetic siliceous glass	Siliceous, with lesser proportions of alkalis, Al_2O_3, and/or other substances	Glass
The most important deleteriously alkali-reactive rocks (that is, rocks containing excessive amounts of one or more of the substances listed above) are as follows:		
Opaline cherts	Rhyolites and tuffs	Opaline concretions
Chalcedonic cherts	Dacites and tuffs	Fractured, strained, and limestone-filled quartz and quartzites
Quartzose cherts	Andesites and tuffs	
Siliceous limestones	Siliceous shales	
Siliceous dolomites	Phylites	
NOTE: A rock may be classified as, for example "siliceous limestone," and be innocuous if its siliceous constituents are other than those indicated above.		

Table 2.2 Example of reactive rocks with mineral, chemical and physical properties (ACI Committee 201, 1991)

The aggregate types which have been documented being alkali reactive based on several studies in the field and laboratory are composed of pure SiO_2 like opal, flint, tridymite or composed of silicates such as andesite, industrial glasses, rocks like rhyolite and greywacke. All of these aggregates classified as alkali reactive have some properties that are common; all these aggregates have SiO_2 as their major component and their structures are either open structure or disturbed structure, some examples of this are industrial glass, tridymite, opal, etc. Also, all these aggregate's external and internal surfaces are covered with silanol. Examples of some reactive rocks are shown in Table 2.2. It should however be noted that silica is present in many types of rock, but not all types of silica reacts extensively with the concrete's pore solution. Therefore, not all aggregates that contain silica and react with a high alkali concrete's pore solution will lead to a deleterious alkali silica reaction.

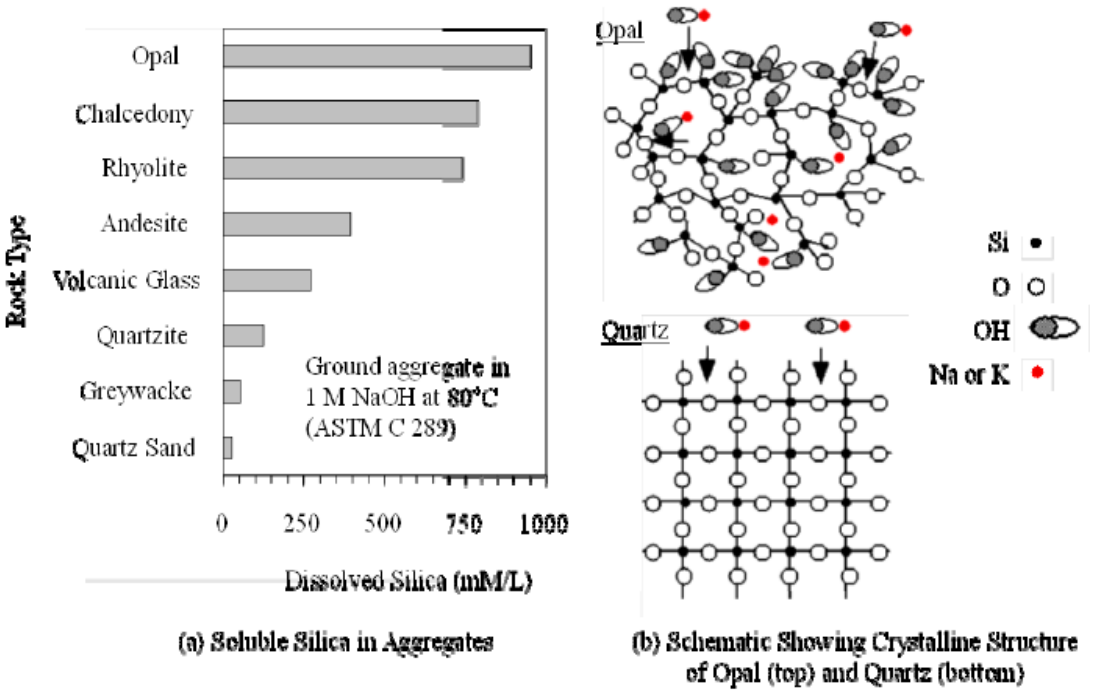


Figure 2.8 Structure and solubility of quartz and opal (AAR Fact book, 2013)

For example, quartz though it will react with the alkali pore solution of concrete it will never produce a damaging alkali silica reaction no matter the concrete's alkali content. While in other hand, though mineral opal and mineral quartz have similar chemical characteristics, the mineral quartz is stable in concrete's pore solution while mineral opal is not and will react with a high alkali pore solution of concrete to produce a deleterious alkali silica reaction. The difference in the solubility of these two minerals and also their crystalline structure as shown in figure 2.8b will help to understand why they react differently. Figure 2.8a shows relative reactivity based on dissolved silica.

2.5.2 CHEMISTRY OF ALKALI SILICA REACTION

To have a better understanding of this reaction between a reactive aggregate and the alkali pore solution of the concrete, it is necessary to know the composition of the aggregate and alkali pore solution, the environment in which the reaction occurs, the type of ions present, properties of the ions such as concentration and also the composition of the product formed as a result of the reaction. All these factors can be designated as the chemistry behind the reaction.

When there is hydroxyl ion (OH^-) in high concentration in concrete, the silica from the aggregate starts dissolving by the neutralization of the silanol group after which it is attacked by the siloxane group. The surface of a siliceous mineral grain in an alkali solution contains the Si-OH group and it ionizes as follows;

$$\equiv Si\text{-}OH + OH^- \rightarrow Si\text{-}O^- + H2O \qquad \text{Eq. 2.3}$$

$$\equiv Si\text{-}O\text{-}Si\equiv + 2OH\text{-} \rightarrow 2Si\text{-}O^- + H2O \qquad \text{Eq. 2.4}$$

The structure of the original silica is shown in figure 2.9. As the reaction occurs, there's a continuous breakdown of the structure as a result of the breakdown of the siloxane bridges as observed in figure 2.10. The dotted lines shown in figure 2.10 show the dissolution of silica at the interface that exists between water and silica. The Silica (SiO_2) is made of mainly siloxane groups represented chemically as ≡Si-O-Si≡.

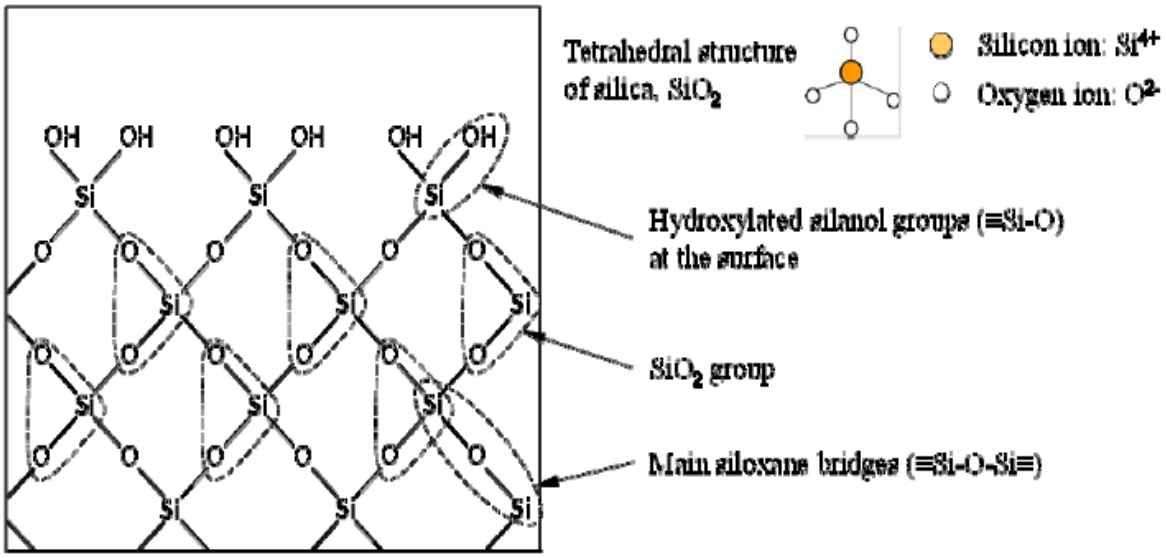


Figure 2.9 Structure of silica (AAR Fact book, 2013)

As the structure breaks down, the positive charged ions such as sodium (Na^+) and potassium (K^+) which are found in surplus in the pore solution of concrete attracts the negatively charged Si-O- ions. The product of this initial stage of the chemical reaction is a solution or gel of alkali-silicate which depends on the amount of moisture available. If there is sufficient moisture available, a solution of alkali silicate is formed; if lesser amount of moisture is available, a gel of alkali-silicate is formed. But if calcium is present, the product formed is just alkali-silicate gel ($CaO\text{-}Na_2O/K_2O\text{-}SiO_2\text{-}H_2O$) which is formed as a result of the precipitation of silica from the solution. The alkali

silicate gel is made up of mainly silica, sodium and potassium with traces of calcium. The properties and characteristics of the reaction product has been found to vary widely (Moranville-Regourd, 1989).

The dissolution of silica in the solution leads to the reduction in the hydroxyl ion (OH^-) concentration. If only silica (SiO_2) and alkali hydroxide are primarily present in the solution, there would be continuous dissolution of the silica until the solution reaches a concentration where there is equilibrium between silica and the pH as shown in figure 2.11. From figure 2.11, it will be observed that the concentration of silica is dependent on the initial ratio of SiO_2/Na_2O.

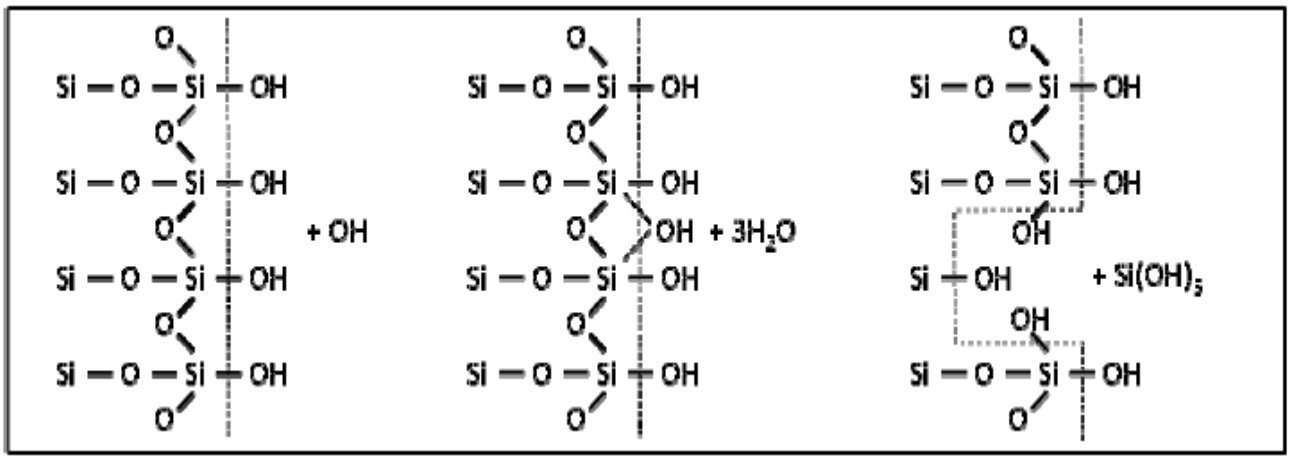


Figure 2.10 Attack of hydroxyl ions on silica which leads to its dissolution (Iler 1979, Urhan 1987)

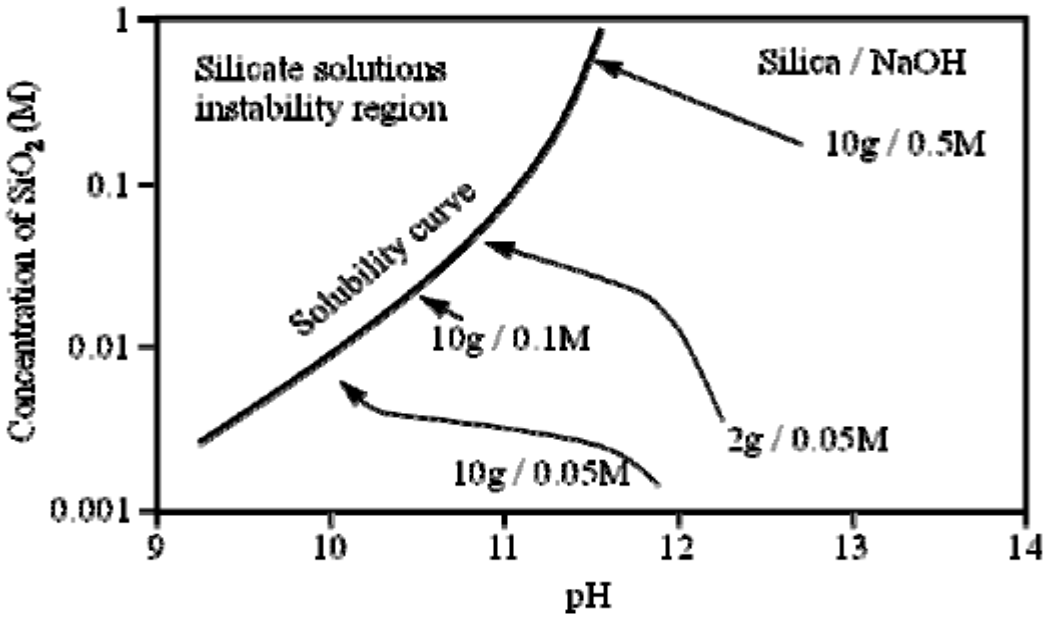


Figure 2.11 Silica-pH equilibrium curve (Dent et al, 1981)

For concrete and mortar, the equilibrium in concentration between silica and pH is achieved slowly because the reaction is more complex and because of the availability of more calcium which leads to reduction in the concentration of silica and a pathway for more hydroxyl ions.

2.5.3 ALKALI SILICA REACTION ENVIRONMENT

The environment inside a concrete is very complex and a knowledge about the concrete microstructure helps to understand it more. When the concrete is mixed, i.e. mixing of aggregates and cement paste, a liquid which is alkali in nature surrounds the aggregates; this pore liquid composition affects the rate and extent in which the alkali silica reaction occurs.

Following the development of the pore solution with time, at about 8 hours after casting, the pore liquid of concrete contains a mixture of Ca^{2+}, K^{+}, OH^{-}, and SO^{2-}_{4} ions (Chatterji et al, 1992). The concentration of Ca^{2+} and SO^{2-}_{4} drops quickly while those of the remaining ions increases sharply,

therefore the concrete pore solution of concrete is converted to alkali hydroxide solution (Diamond, 1983). If there is no external moisture ingress, the alkali content of the cement is the alkali hydroxide concentration present in that concrete.

2.5.4 COMPOSITION OF REACTION PRODUCTS

When alkali silica reaction occurs, a product is formed which is a hygroscopic gel. The analyses of this gel have shown that the gel consist of alkali silicate hydrates with some $CaCO_3$ present in it. These gels have been obtained from the surfaces of structures that have been severely damaged by alkali silica reaction. Though these gels obtained have been affected by carbonation, they were still able to be analyzed at the early stage by the help of the electron probe micro analytical technique (Chatterji et al, 1992, Regourd, 1989, Kawamura et al, 2003). In the studies these researchers carried out, it was shown that the products of the reaction are complex; composed of lime, alkali, silica and water at different compositions. When the gel which is the product is dry, the product might be composed of 20% CaO, 20% alkali oxide and the remaining approximately 60% is SiO_2.

2.5.5 MECHANISM OF ASR DETERIORATION IN CONCRETE

Just the reaction between the alkali from high alkali cement and silica from reactive aggregate does not deteriorate the concrete; it's when the silica gel formed absorbs moisture and expands that causes deterioration. Figure 2.12 shows a thin section of a concrete undergoing alkali silica

reaction; it can be observed that the aggregate (flint) has undergone the reaction process and the alkali silica reaction has led to the expansion and cracking of the aggregate itself and extending into the cement paste around the aggregate. The generalized sequence for alkali silica reaction is shown in figure 2.13.

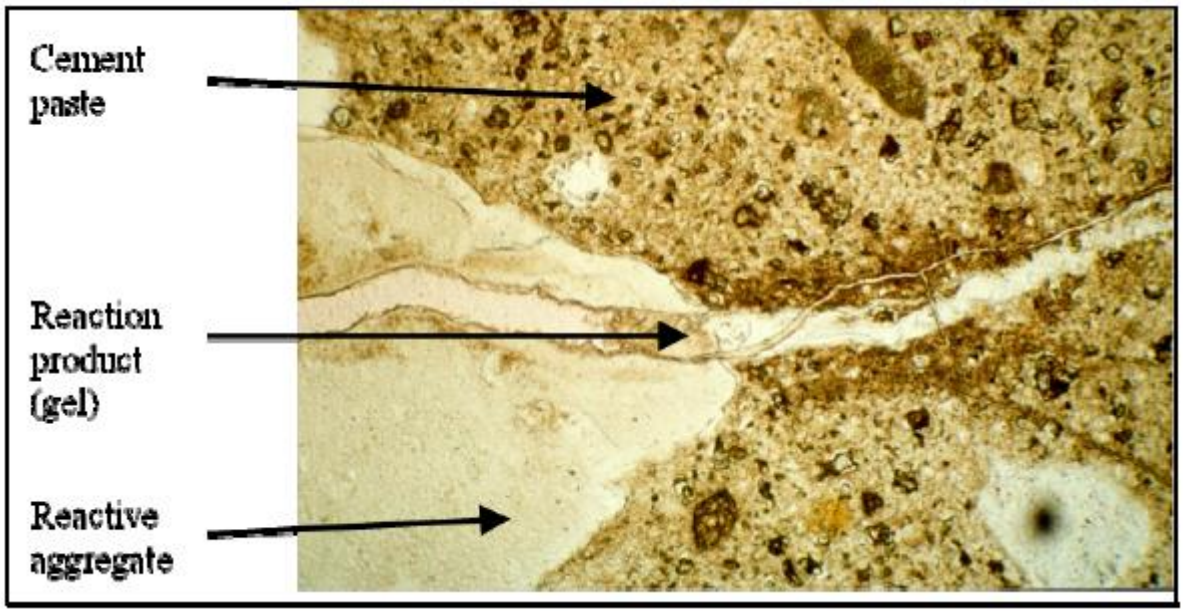


Figure 2.12 Thin section of a concrete affected by ASR viewed under microscope (AAR Fact book, 2013)

Although there has been a general acceptance about the chemical processes in alkali silica reaction, there has not been a consensus in terms of the mechanism of the reaction. Few examples of researchers with different views are; Hansen (1994), McGowan and Vivian (1952), Powers and Steniour (1952). While McGowan and Vivian (1952) proposed that the expansion mechanism is based on the gel being hygroscopic and being able to absorb moisture, thereby increasing in

volume causing extensions and cracking in concrete, Hansen suggested that the mechanism of ASR is as a result of the increase in hydrostatic pressure being put on the cement paste as a result of the osmotic pressure cell formed. For Powers and Steniour (1952), they suggested that the product formed from the alkali silica reaction is a semi-permeable membrane and that depending on whether the product is in liquid or solid form; imbibition and osmotic pressures may be generated. Despite the lack of consensus about the mechanism of ASR, the mechanism in which it causes deterioration can be summarized in four steps as follows (Ichikawa et al, 2007):

i) The siliceous aggregate is being attacked by the alkali solution thereby forming a silicate gel that is viscous in nature.

ii) The alkali in the solution causes the Ca^{2+} ions to dissolve into the pore water of the concrete, then the calcium reacts with the viscous gel formed to make it hard C-S-H.

iii) The extra alkali in the concrete due to high alkali cement or other sources changes the remaining siliceous mineral into a gel that is bulky called the alkali silicate gel, the expansion caused by this change only occurs in the aggregate.

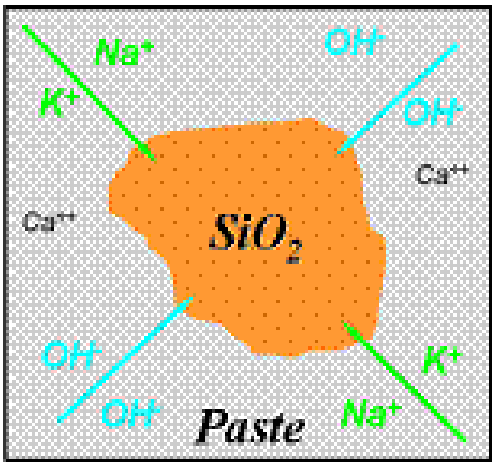
iv) When the pressure as a result of this change can no longer be accommodated by the aggregate, the pressure cracks the aggregate and the cement paste that surrounds it.

Studies have shown that when calcium is not present in the pore solution, the alkali hydroxide causes dissolution of the silica without causing any deteriorating effect on the concrete (Diamond 1989, Thomas 1998). But if high concentration of calcium exists in the pore solution; it guards against the movement of silica away from the aggregate in which it is present (Chatterji 1979; Chatterji and Clausson-Kass 1984). Calcium aids the exchange of alkalis in the product of the reaction by a process called ‘alkali recycling’ which leads to more alkalis being produced in the pore solution available for more reaction (Hansen 1994, Thomas 2001).

(a)

Concrete pore solution is dominated by Na, K & OH (with minor amounts of Ca).

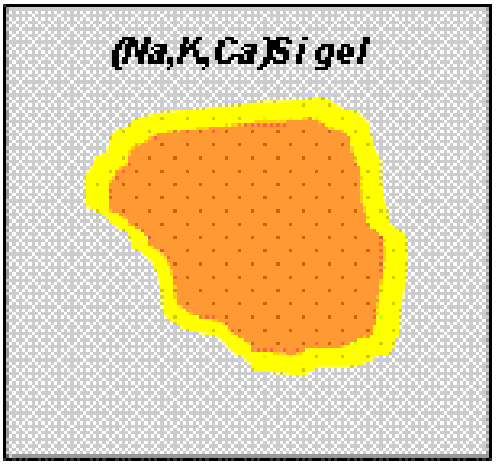
If the silica in the aggregate is reactive – the OH and then the Na & K will react with the SiO_2



(b)

The product of the reaction is in alkali-silica gel composed of Na, K, Ca & Si

The gel forms around and within the aggregate



(c)

The gel imbibes water from the surrounding cement paste

The gel expands

Eventually the swelling pressures may exceed the tensile strength of the surrounding paste and cause expansion & cracking of the concrete

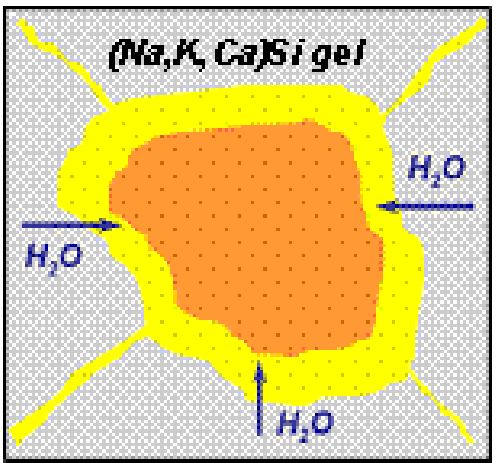


Figure 2.13 Sequence for Alkali Silica reaction (AAR Fact book, 2013)

There are studies that have shown that there is an exceptional expansion that occurs when calcium is available as calcium hydroxide ($Ca(OH)_2$), in the pore solution the calcium hydroxide acts as a reservoir for hydroxyl ions which helps to achieve a high level of hydroxyl ion in the solution (Wang and Gillot, 1991).

2.5.6 ALKALI SILICA REACTION TEST METHODS

There have been several tests methods that have developed since this reaction was discovered by Stanton in the late 1930s in California. Several studies have proposed ways in which aggregates can be tested to see if they are prone to the reaction and to find ways to avoid the reaction occurring in new concrete works. One of the first tests developed by Stanton is the current ASTM C 227. Other tests methods have been accepted and standardized while some have been rejected due to their inability to detect reactive aggregates accurately.

One of the major limitations of the rejected tests and some of the ones still in use is the results are not consistent with what is observed in the field. Table 2.3 shows the most common test methods used to determine the reactivity of aggregates and Table 2.4 shows tests for evaluating preventive measures for ASR. More focus will be placed on two main tests which have highest credibility; the accelerated mortar bar test (AMBT) and concrete prism test (CPT), these methods have also been standardized by American Association of State Highway and Transportation officials (AASHTO) PP65-11.

Test Method	**Comments**
ASTM C 295: Standard Guide for Petrographic Examination of Aggregates for Concrete	• Useful evaluation to identify many (but not all) potentially reactive components in aggregates. • Reliability of examination depends on experience and skill of individual petrographer. • Results should not be used exclusively to accept or reject aggregate source – findings best used in conjunction with other laboratory tests (e.g., AASHTO T 303 and/or ASTM C 1293).
ASTM C 289: Standard Test Method for Potential Alkali-Silica Reactivity of Aggregates (Chemical Method)	• Aggregate test in which crushed aggregate is immersed in 1M NaOH solution for 24 hours – solution is then analyzed for amount of dissolved silica and alkalinity. • Poor reliability. • Problems with test include: – Other phases present in aggregate may affect dissolution of silica (Bérubé and Fournier 1992). – Test is overly severe, leading aggregates with good field performance to fail the test. – Some reactive phases may be lost during pretest processing.
ASTM C 227: Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)	• Mortar bar test (aggregate/cement = 2.25), intended to study cement -aggregate combinations. • Specimens stored in high-humidity containers at 38°C. • Several reported problems with test, including excessive leaching of alkalis from specimens.
AASHTO T 303 (ASTM C 1260): Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)	• Mortar bar test, originally designed to assess aggregate reactivity. • Bars are soaked in 1N NaOH solution for 14 days. • Accelerated test suitable as screening test, but because of severity of test, it should not be used, by itself, to reject a given aggregate. If aggregate is tested using both AASHTO T 303 and ASTM C 1293, the results of ASTM C 1293 should govern.
ASTM C 1293: Standard Test Method for Concrete Aggregates by Determination of Length Change of Concrete Due to Alkali-Silica Reaction	• Concrete prism test, generally regarded as best indicator of field performance, is conducted at high humidity (close to 100%) at 38° C. • Uses high-alkali cement (raised to 1.25% Na_2O_e), with a cement content of 420 kg/m^3. • Developed as aggregate test (using non-reactive fine aggregate to test reactivity of coarse aggregate, and vice-versa). • Test requires one year for completion – this long duration limits its use by many agencies and owners. • Cannot be used to determine the alkali threshold for a given aggregate due to leaching of alkalis from the prisms during the course of the test.

Table 2.3. Common test methods used to determine aggregate reactivity (AAR Fact book, 2013)

Test Method	Comments
ASTM C 441: Standard Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction	• Mortar bar test, intended to assess effectiveness of SCMs in reducing ASR expansion. • Test uses high-alkali cement and Pyrex™ glass. • Test not very reliable because of the use of Pyrex glass, which is sensitive to test conditions and contains alkalis that may be released during the test. Test does not correlate well with data from concrete mixtures containing natural aggregates (Bérubé and Duchesne
ASTM C 1567: Standard Test Method for Determining the Potential Alkali-Silica Reactivity of Combinations of Cementitious Materials and Aggregate (Accelerated Mortar-Bar Method)	• Mortar bar test, originally designed to assess aggregate reactivity. • Bars are soaked in 1N NaOH solution for 14 days. • Should only be used for aggregates for which a reasonable correlation between AASHTO T 303 (ASTM C 1260) and ASTM C 1293 has been established (see AASHTO PP65-11 for details). • ASTM C 1567 can be modified as per AASHTO PP65-11 to evaluate lithium nitrate-based admixtures, although ASTM C 1293 is deemed to be a more effective test method for determining
ASTM C 1293: Standard Test Method for Concrete Aggregates by Determination of Length Change of Concrete Due to Alkali-Silica Reaction	• Modified version of concrete prism test, allows for testing SCMs, blended cements, and lithium admixtures. • Test duration is two years, with expansion limit of 0.04 percent.

Table 2.4. Test methods for evaluating preventive measures (AAR Fact book, 2013)

2.5.6.1 ASTM C 1260 (CSA A 23.2 – 25A) (Accelerated Mortar Bar Test)

The accelerated mortar bar test is a standardized test method to determine the potential alkali reactivity of aggregates to be used in concrete. This test method employs the use of mortar instead of concrete, high concentrations of alkali and high temperatures to allow the detection of aggregate reactivity within 16 days. It was developed at the National Building Research Institute in South Africa by Oberholster and Davies (Oberholster et al, 1986), this test was accepted and adopted into different agencies and countries such as CSA in Canada and AASHTO and ASTM in United States.

This test requires making mortar bar samples using a water ratio of 0.47 and a mold of 25mm x 25mm x 285mm. After the mortars are cured in a moist room for approximately 24 hours, they are soaked in a water bath and placed in an oven at 80°C for approximately 24 hours. After this, zero readings taken and the mortar bars are then soaked in 1 N NaOH solution and placed in the oven at 80°C. Measurements are taken daily for 14 days with an optional additional two weeks if testing for the effectiveness of a supplementary cementing material to curb ASR. The average difference between the zero readings and readings of the specimen at each period is calculated to the nearest 0.001% of the effective gauge length and recorded as the expansion of the specimen for that period. Though the ASTM C 1260 and CSA A23.2-25A are similar, the expansion limit at the end of the 14 days are different and shown below

ASTM C 1260 expansion limit:

< 0.10% is considered innocuous

0.10 to 0.20% is considered potentially reactive

> 0.20% is considered reactive

CSA A23.2-25A expansion limit:

> 0.15% (0.10% for limestone) is considered reactive

< 0.15% (0.10% for limestone) is considered non-reactive

2.5.6.2 ASTM C 1293 (Concrete Prism Test)

The concrete prism test is accepted to be more accurate than the ASTM C 1260, but the major challenges of this test are the long duration of testing (1 year or longer) and leaching of alkali during test period which result in prisms not expanding the way they should have expanded. It has been reported that almost 35% of the total alkali within concrete leach out during this process (Thomas et el, 2006) leading to samples not expanding as they ought to if the alkali did not leach out, and almost 20% leached out during the first 90 days as shown in figure 2.14.

This test uses concrete made with 420 kg/m^3 cement content and an alkali content between 0.8% and 1.0%, additional sodium hydroxide is added to the mixing water to boost the alkali content to 1.25% by mass of the cement. The prisms are cast and cured for approximately 24 hours at 23°C, after which they are stored over water at 38°C for a duration of 1 year if no SCMs were used and 2 years when SCMs are incorporated in the samples. Measurement of expansion is performed at regular intervals to calculate the expansion at a particular period in time. The limit for this test is 0.04% expansion at 1 year (or 2 years with SCM).

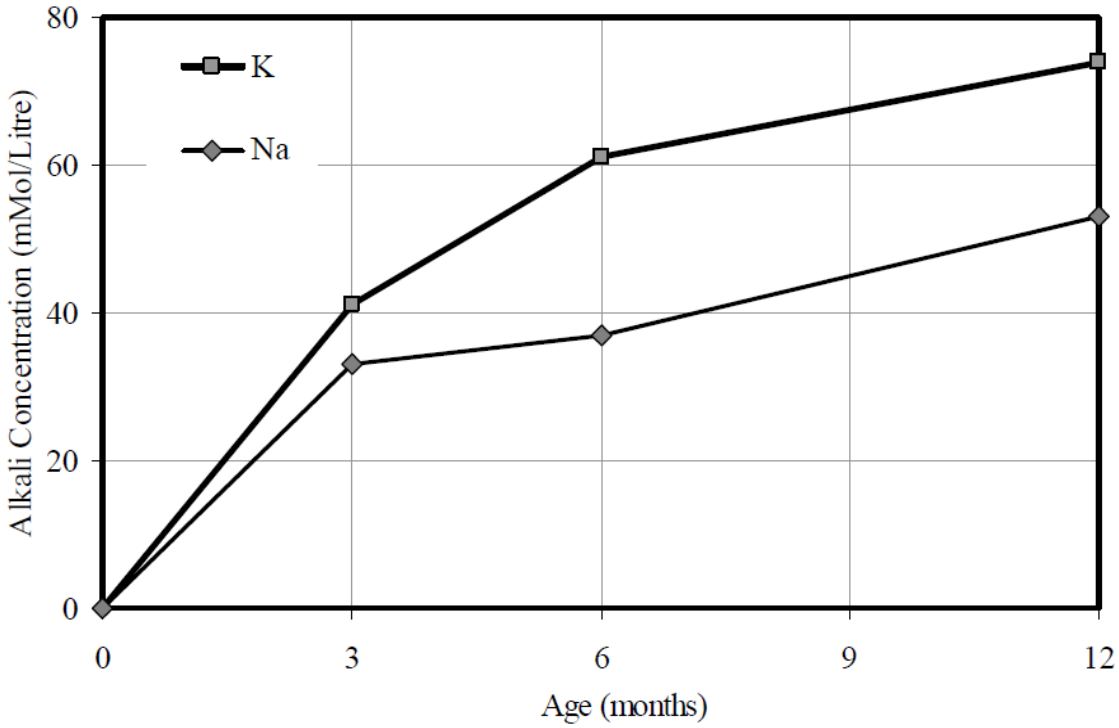


Figure 2.14 Concentration of alkalis measured in the water at the bottom of an ASTM C1293 Container as a function of time (Thomas et al. 2006)

Generally, to test if an aggregate is susceptible to ASR, the flow chart presented in figure 2.15 can be followed which provides a step by step procedure to determine if aggregates to be used in new construction are susceptible to ASR starting from observing if the aggregate has adequate field performance based on existing structures exposed to similar environment as the new structure. Based on the answer; whether to test the aggregate would be determined and also to determine if the reaction can be mitigated if the aggregates are found to be reactive.

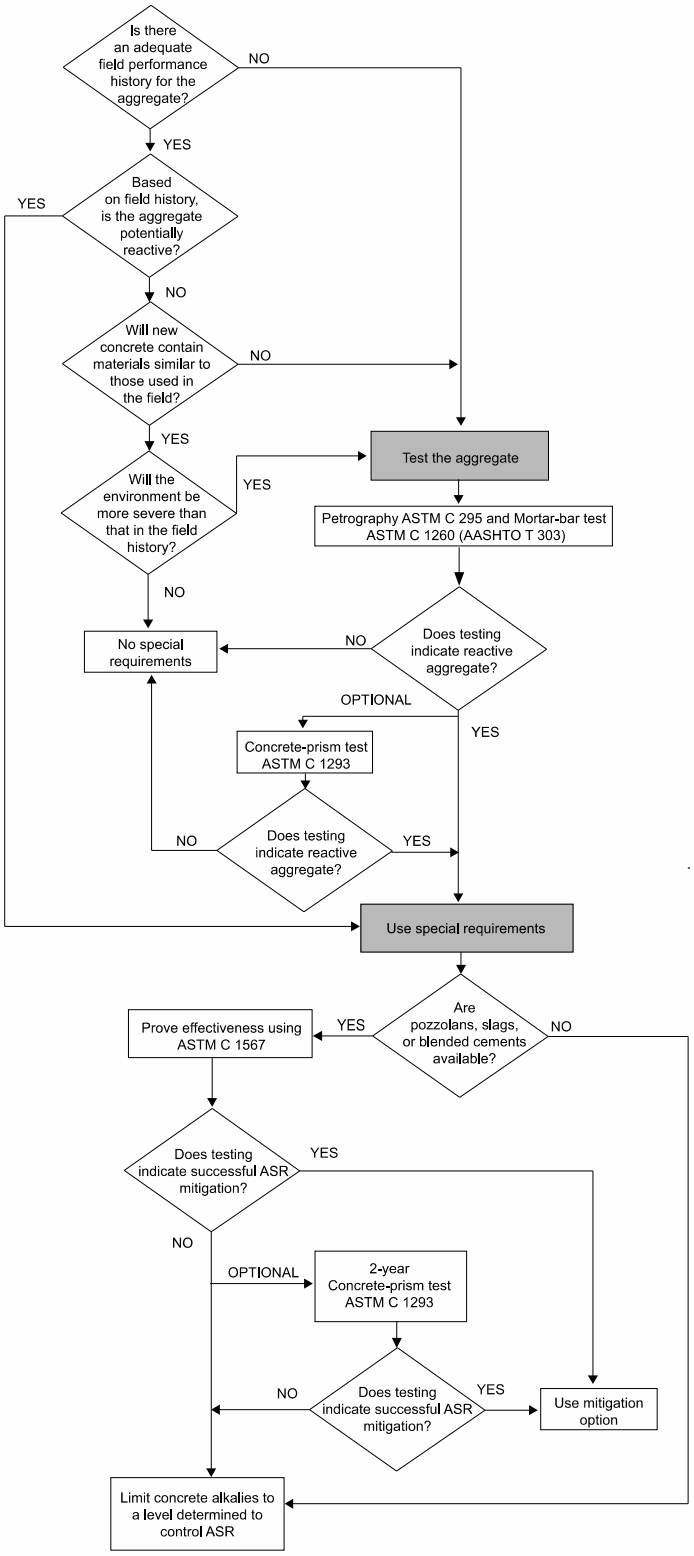


Figure 2.15 ASR Control Flow Chart (www.cement.org)

2.6 MOISTURE IN CONCRETE

In this thesis, the moisture in concrete is also be referred to as water, but it should be noted however that water can exist in different forms depending on the environmental condition in which it is found. Literally not seeing liquid water does not mean that moisture is not present; it might be present in vapor form. Moisture existing in different states and the complex microstructure of concrete has made its measurement in concrete very complex and there exists no definite method for measuring moisture in concrete.

It has been observed from different papers reviewed that there has been a high demand to understand the moisture content inside concrete as this has been found to affect not only the properties of concrete but also its deteriorating effect especially when focusing on the corrosion of reinforcing steel and durability of concrete in general.

2.6.1 FORMS OF MOISTURE IN CONCRETE

As discussed earlier, water in concrete is capable of existing in different forms inside concrete; the form in which it exists depends on the level of which it can be removed from the cement paste. Water referred to here as moisture can exist in concrete in four different forms; capillary water, adsorbed water, interlayer water and chemically combined water. These exist within and around the calcium silicate hydrate are shown in figure 2.16 and are further discussed.

2.6.1.1 Capillary Water

Capillary water is the form of water in concrete that is available in voids larger than approximately 50Å. It can be referred to as the form of water existing in the concrete that are not affected by the attractive forces exerted by the concrete. This type of water in concrete can be further divided into two which are; the free water which is present in large voids greater than greater than 50nm (0.05µm) and the other type of capillary water is that present in small capillaries (5 to 50nm). The first type is called free water because its removal from the cement paste does not cause any volume change while the removal of the water in smaller capillaries can cause shrinkage of the cement paste system because it is held by capillary tension.

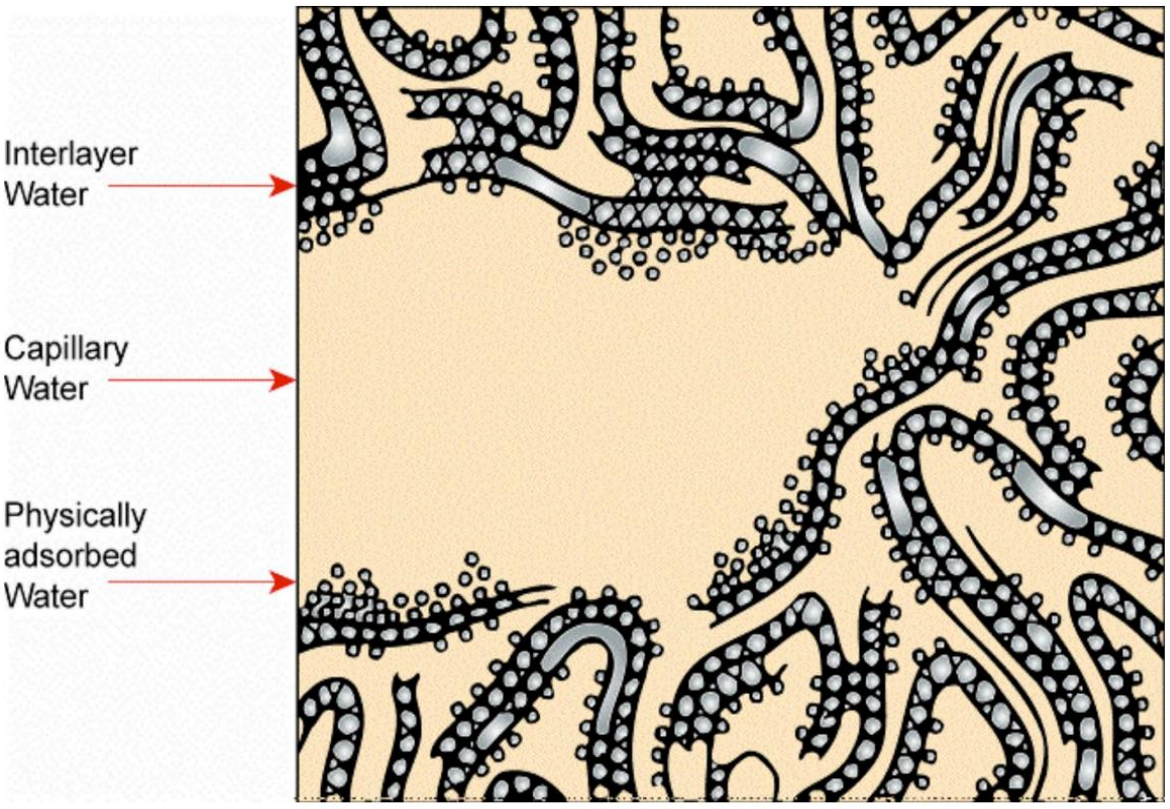


Figure 2.16 Model for types of water in concrete (Mehta et al, 2006)

2.6.1.2 Adsorbed Water

The water in concrete close to the solid surface is called adsorbed water, molecules of water are always adsorbed on the surface of solids under the influence of attractive forces. For individual water molecules, the bond energies decrease as it move away from the surface of the solid. A significant amount of adsorbed water can be removed if a hydrated cement paste is dried to a low relative humidity (like 30 percent), shrinkage of the hydrated cement paste also occurs as a result of the loss of a significant amount of the adsorbed water.

2.6.1.3 Interlayer Water

In between the C-S-H structure existing inside concrete, the water is called the interlayer water and this type of water can be eliminated only when the concrete is dried strongly because the molecules of water is held strongly by hydrogen bond. Drying of this water will lead to shrinkage of the C-S-H structure.

2.6.1.4 Chemically Combined Water

As the name implies, chemically combined water is involved in the main chemical process inside concrete which is hydration. When concrete is dried, compared to other water existing in concrete, the chemically combined water is not lost, it can only evolve when the hydrates decompose at very high temperatures.

2.6.2 MEASURING MOISTURE IN CONCRETE

In most cases, the moisture in concrete is not uniformly distributed and varies with exposure time which makes the moisture content difficult to determine. The ideal way to measure moisture in concrete should be able to quantify the amount of all the types of water existing in concrete, this ideal method should also be sensitive to changes in the moisture state inside a concrete.

Moisture content of a concrete is the total amount of moisture existing in concrete either as water vapour or liquid water and its always expressed as a percentage of the concrete's mass. As already mentioned, moisture can be present in smaller pores and capillary pores in concrete and can exist as vapour or liquid water when the pores are saturated and the concrete is wet. There are several methods for measuring moisture in concrete such as dielectric properties, neutron scattering, relative humidity, thermal properties, resistivity, destructive samples (Parrot, 1990), some of these methods will be further explained. Table 2.5 shows some techniques of measuring moisture in concrete and their characteristics.

Method	*Geometry*	*Approximate sampled Volume/depth*	*Accuracy*[1]	*Specific to water?*	*Water measured*			*Moisture gradients*	*Cost*	
					Free	*Absorbed*	*Bound*		*Equip*	*Labour*
Drill or cut and standardized drying	Borehole	~1 to 5 cm^3	High	No	✓	✓	✓	✓	Low	High
Relative humidity	Borehole	~2 to 10 cm^3	Moderate	Yes	✓	✓	–	✓	low	Moderate
Resistivity	Surface	~100 cm	Low	No	✓	✓	–	–	Low	Low
	Embedded electrodes	Proportional to electrode spacing	Low	No	✓	✓	–	✓	Low	Moderate
Dielectric properties	Surface	~5-15 cm	Moderate	Yes	✓	–	–	–	High[2]	Low
	Prismatic (attenuation)	Prism thickness	High	Yes	✓	–	–	✓	High[2]	Low
Thermal properties	Embedded probe	~1 cm or more	Low	No	✓	✓	–	✓	Low	Low
Infrared properties	Surface probe	~1mm or less	High	Yes	✓	–	–	–	High	Low
	Surface thermography	~1 mm or less	Low	No	✓	✓	–	–	High	Low
Neutron scattering	Surface	~100 cm	Moderate	No	–Total water–			–	High	Low
	Borehole	~100 cm	Moderate	No	–Total water–			✓	High	Moderate

1 Assuming calibration data is available for test material
2 Could be reduced if commercial equipment was available

Table 2.5 Methods of Measuring Moisture in Concrete and their characteristics (Hilsdorf et al, 2004)

Gravimetric Moisture Content Test

This is a simple destructive test carried out on concrete to determine its moisture content, the procedure for this test involves taking pieces or core samples from the structure to be tested and drying the sample until a constant mass is achieved. The moisture content is calculated by calculating the difference between the final and initial mass of the sample taken. This test is preferred to calcium chloride test because it gives a good representation of moisture content of the full thickness of the sample taken and not just the surface and its able to measure moisture content

of concretes with high moisture content (Jana 1996). The moisture content can also be expressed by volume as the difference in the total volume of concrete sample and the volume of water (DeAngelis 2007).

Infrared Absorption

Electromagnetic radiation at some specific wavelengths is absorbed by water. Porous material's water content can be determined by observing absorption of water at different wavelengths. To simplify the way to use infrared absorption to measure water content, the source of infrared radiation and detector has to be placed at a distance of 50mm to 300mm, and measurements are always carried out in reflection mode rather than transmission mode because infrared beam can only pass through a thin layer of the surface of the sample (Parrot, 1990). Cornell et al (1971) found a good correlation between the actual moisture content measured gravimetrically and infrared absorption as shown in figure 2.17. One of the disadvantages of this method is that variation in colour and texture of sample surface can affect the results (Cornell et al, 1971). This method requires costly equipment and is not suitable for insitu measurements.

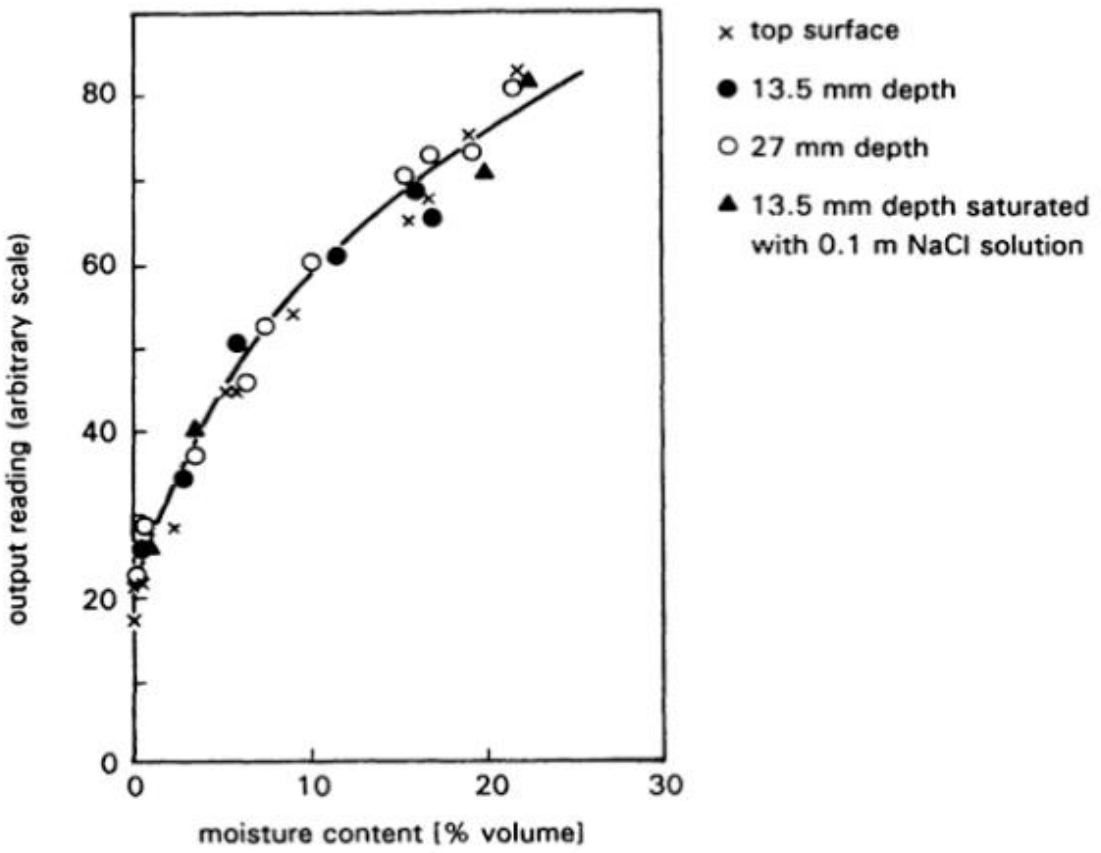


Figure 2.17 Moisture content determination for 1:1:6 cement: lime: sand mortar samples using infrared absorption method (Cornell et al, 1971)

Electrical Resistivity

Porous material's electrical resistivity decreases with increase in moisture content as a result of the resistivity of the pore solution being extremely lower than the resistivity of the solid component of the material which makes it possible to use resistivity to measure moisture content of a concrete. Various studies have shown that loss of pore water in mortar and concrete leads to an increase in its resistivity and this is more noticed in concrete and mortar with lower water content. Figure 2.18 shows the variation of loss of moisture with resistivity.

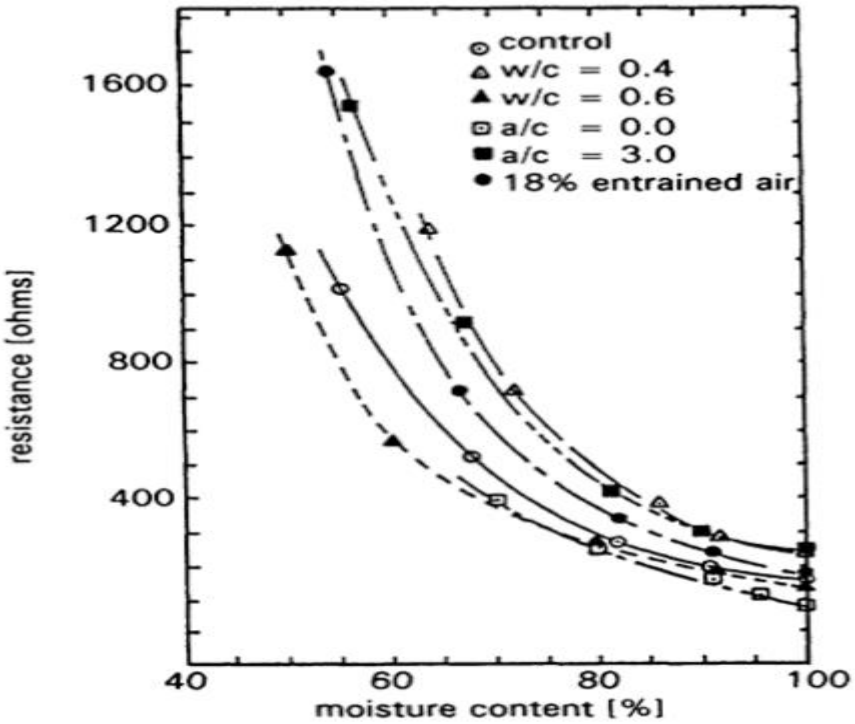


Figure 2.18 Variation of resistance with moisture content for various water to cement ratio (Parrot 1990)

The result obtained by using resistivity to measure moisture content in concrete is affected by the composition of pore fluid, type of cement used, temperature, cement hydration and water to cement ratio. Parrot (1990) concluded that good representation of moisture content in concrete can be obtained using resistivity method if there's detailed knowledge about moisture gradient and careful calibration is done for each condition of test. Figure 2.19 shows a typical setup for measurement of resistivity of a concrete sample using a device with power source and four electrodes.

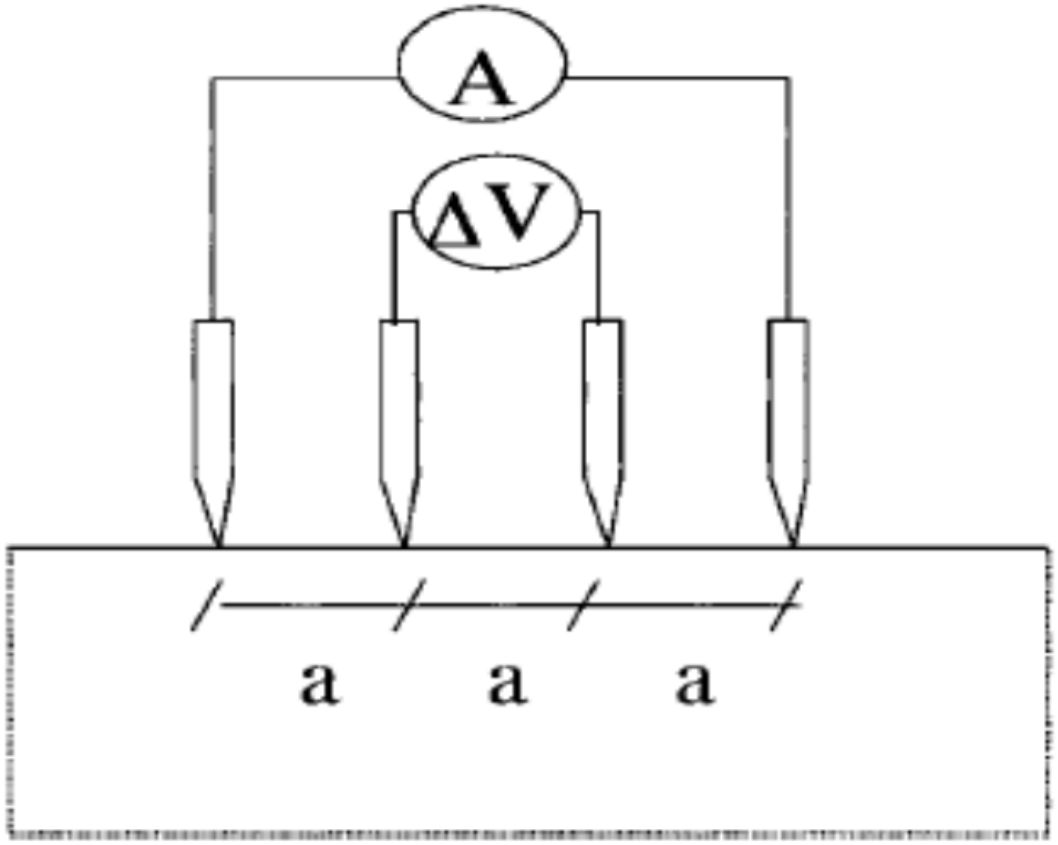


Figure 2.19 Setup for resistivity measurement on a sample (Polder, 2001)

Calcium Chloride Test

This test was developed in the United States to measure the moisture content of concrete floors and the procedure covered in ASTM F1869-04, the test measures the moisture content by measuring the rate of vapour emission from the surface of the concrete. The test is carried out by covering about 210mm x 210mm area of concrete surface for a duration between 60 to 72 hours. Approximately 16g of anhydrous calcium chloride is placed below the cover to absorb any moisture in terms of vapour emitted from the concrete surface. The rate of vapour emission is calculated from the weight of the test apparatus before and after the test period, the surface area and the duration of test.

Neutron Scattering Method

In this method, fast moving neutrons from a radioactive source are emitted and they are able to penetrate concrete, these neutrons collide with the nuclei of atoms that make up the concrete (Gonzalo 2011). Since water molecules contain hydrogen, the collision of the fast moving neutrons with the hydrogen leads to the production of slow moving neutrons in concrete and these slow moving neutrons can be monitored with the help of a detector that has a slow neutron absorber. To be able to differentiate between chemically bound, absorbed and free water using this technique, high resolution detectors have to be incorporated in the neutron scattering equipment used (Gonzalo 2011). There might be slow absorption of neutrons because of some elements such as calcium, aluminum, and oxygen present in concrete and cement (Parrot 1990). There is a huge health concern with this method has it involves the use of radioactive materials which has made the use only available to trained individuals and the commercial production of the equipment licensed (Parrot 1990).

Thermal Property Method

The same way electrical resistivity can be used to determine the water content of a porous material, knowing its thermal properties can be used to determine its moisture content by determining the thermal conductivity of the material and collating the conductivity with a calibrated data to obtain the moisture content (Parrot 1990).

To get the thermal conductivity of concrete; heaters and small temperature probes are embedded in concrete, then the heater is activated and the thermal response is measured as a function of time.

Concrete's thermal properties are being affected by factors such as aggregate type, air entrainment, aggregate's proportion and concrete's density. (Parrot 1990)

Relative Humidity Measurement

Measurement of moisture content in concrete can also be done by measuring the relative humidity inside concrete. With the advancement of technology, several sensors and probes have been developed to measure relative humidity, but no specific one is designed to measure just relative humidity in concrete as most of the sensors available are used in the food, paper and health industries for quality management.

Human and horse hair are mainly used in making high quality relative humidity sensors (Gonzalo et al, 2011), strain gauge helps to convert the movement of hair to an electrical signal. Other sensors are made from water absorbent polymers and cellulose butyrate that change with changes in relative humidity.

Two major types of sensors used to measure relative humidity exist and can be classified into resistive sensors and capacitive sensors. The resistive sensors measure relative humidity by measuring the electrical resistance over the surface of the polymer as the polymer reacts to moisture content changes, these resistive relative humidity sensors have slow response to changes in relative humidity (Physics 2010). The capacitive relative humidity sensors measure relative humidity by measuring the electrical capacitance and current between the sensor electrodes, these types of sensors need their own supply of power before measurements can be taken (Physics 2010), so they are always connected to a data logger which supplies the power.

Relative humidity sensors are likely to be exposed to harsh environments and can get easily contaminated. So for use in a harsh environment like concrete, the sensors can be protected with a thin layer of silicone or Gore-Tex because they allow flow of water molecules. It should however be noted that these protections will extend the response time of the sensors to relative humidity changes (Physics, 2010)

There is another method that can be used to measure relative humidity called the Wooden Stick Method (Jensen, 2003), this method is mostly used in concrete with high relative humidity. This method is relatively cheap as the kit needed is inexpensive, this technique measures the electrical conductivity between two steel needles that is pressed into a particular wood and the electrical conductivity. The wooden sticks are being calibrated before use to be able to determine the relative humidity measured and adsorption and desorption curves are obtained (Jensen, 2003).

To be able to get accurate relative humidity measurements with this technique, each type of stick used has to be calibrated and curves obtained instead of having a general curve for all sticks. Figure 2.20 shows an example of desorption and adsorption curve obtained for a particular wooden stick and confidence interval of 95% obtained from 112 wooden sticks. It can be observed from the figure that the inclination of the curve is steeper at higher relative humidity which means sensitivity of the wooden stick is improved with higher relative humidity. A typical setup for this method is shown in figure 2.21.

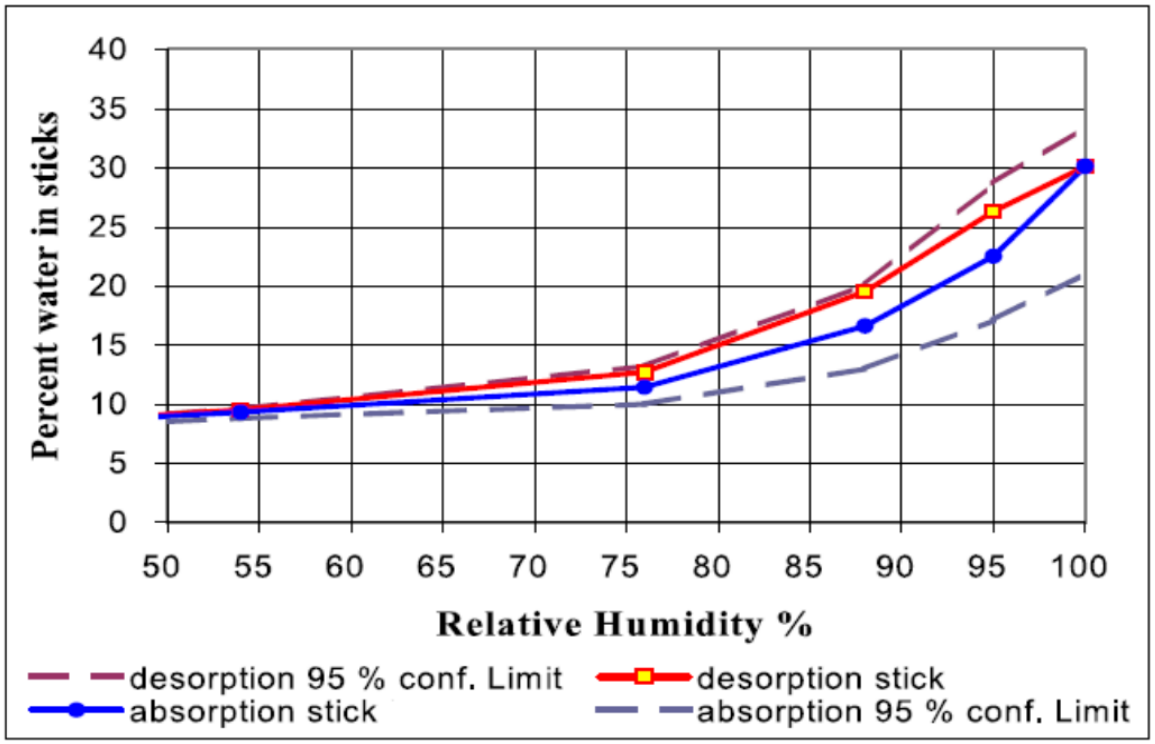


Figure 2.20 Wooden Stick Absorption and Desorption Curve with 95% confidence interval for 112 wooden sticks (Jensen ,2003)

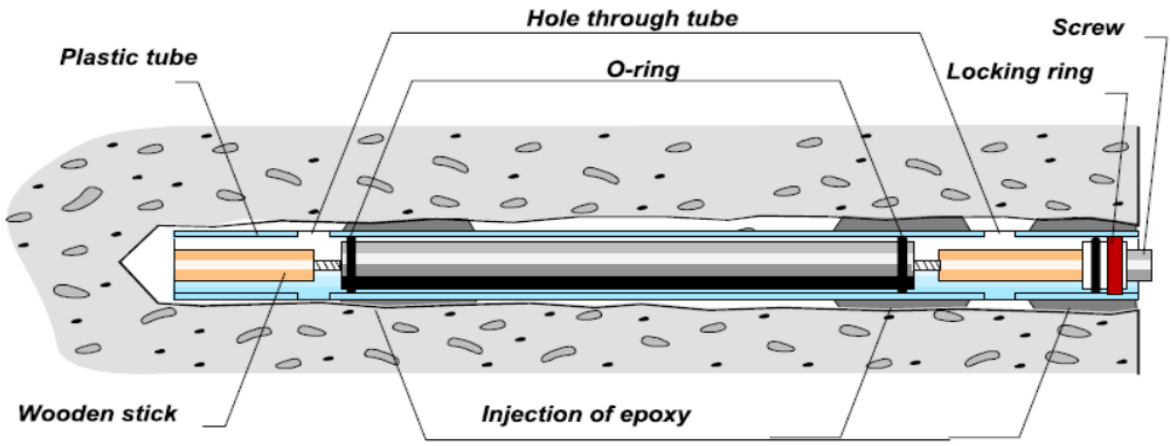


Figure 2.21 Typical setup for Wooden Stick Method to measure relative humidity (Jensen, 2003)

The moisture condition in concrete can be described in two different ways, which are as relative humidity (RH) or as the degree of saturation (Lindgård et al., 2006). It is important to note that the relative humidity is a measure of thermodynamic state of pore water and is not a direct measure of the amount of water (Sellevold et al, 2006). The aggregate porosity and the aggregate moisture state at the time of mixing might significantly influence the relative humidity within the concrete. For example, the use of pre-wetted aggregates with relatively high porosity supply additional water to the cement paste during the curing period and thereby reducing the self-desiccation of cement (Hammer et al., 2004). The measurement of internal relative humidity in concrete has mostly been focused on by researchers in the field of creep and shrinkage of concrete.

2.6.3 RELATIVE HUMIDITY AS AN INDICATION OF MOISTURE

In concrete internal relative humidity is related to moisture, but should not be confused with moisture content as they are two different properties. Relative humidity is a measure of the concentration of water vapor in concrete; while the moisture content is referred to the mass of water in the sample. However, measuring relative humidity in concrete will help to predict and characterize shrinkage, estimate degree of hydration, understand the effect of moisture on corrosion of steel reinforcement, alkali-silica reactivity and sulfate attack and also freeze and thaw susceptibility among others. Many factors influence the relative humidity inside concrete, such as ambient relative humidity, degree of hydration, and internal temperature (Grasley et al. 2006)

From research and several literature reviews, there are several sensors and methods to measure relative humidity in concrete with no specific one outstanding, the advantages of some of these

sensors is that they are able to also measure the temperature. Relative humidity measurements in concrete are affected by changes in temperature (Nilsson, 1980). This effect of temperature is as a result of sensitivity of the hygrometer used and a minor effect of temperature on the sorption isotherm and differences in temperature between the concrete being tested and the probe used (Nilsson (1980). Nilsson (1980) advised that the probe to be used to measure relative humidity in concrete should be in temperature equilibrium with concrete before measurements are taken so as to eliminate the effect of temperature on the measurements obtained, he also suggested that there is variation of 0.1 to 0.5% per °C for relative humidity measurements.

Hydroscopic materials are generally used in making the sensing elements of the relative humidity sensors, this material measure the changes in capacitance, resistance or length, these sensors are available in the market and some are portable enough to be used to measure the conditions inside concrete. It was reported by Monfore (1963) that the repeatability of relative humidity measurement was up to ±2%, this was also agreed to by Parrot (1990) which got a standard deviation of around 2.5% for relative humidity measurement in similar concrete samples.

2.7 MOISTURE AND ALKALI SILICA REACTION

A sufficiently high amount of moisture has to be present before alkali silica reaction becomes a deteriorating element in concrete. Although alkali from concrete's pore and silica in aggregates reacts together to form a silica gel, this reaction does not contribute any deteriorating effect at this time; it is when the silica gel with its hydroscopic properties absorbs moisture from the

environment making it become larger and posing tension in the concrete leading to expansion and cracking of the concrete when its tensional limit is exceeded. Therefore, moisture has to be present before alkali silica reaction becomes a threat to the concrete. The moisture that influences ASR is the available water within the affected material near the reaction sites (Poyet et al. 2006).

Moisture plays two major important roles in ASR; it serves as a transport medium for the ions involved in alkali silica reaction, and serves as a reactant in the reaction. These roles of moisture in alkali silica reaction makes it therefore a matter of priority in alkali silica reaction. It has been shown that the level of available water is directly proportional to the expansion on mortar samples (Vivian, 1981). Several researchers showed that drying the samples affected by alkali silica reaction can stop the reaction (Olafsson, 1986, Tomosawa et al, 1989 and Kurihara et al, 1989). Table 2.6 and figure 2.22 shows the summary of the outcome of their results and conditions employed.

	Author		
Measurement	Olafsson	Tomosawa et al.	Kurihara et al.
Temperature	23 and 38°C	40°C	40°C
Sample shape	Prismatic	Prismatic	Unknown
Dimensions	Unknown	7.5*7.5*40 cm	Unknown
Material	Mortar	Concrete	Mortar
Duration	22 months	52 weeks	12 weeks
Results	Expansion	Expansion and mass	Expansion and water content

Table 2.6 Test Conditions obtained by various researchers (Poyet et al, 2006)

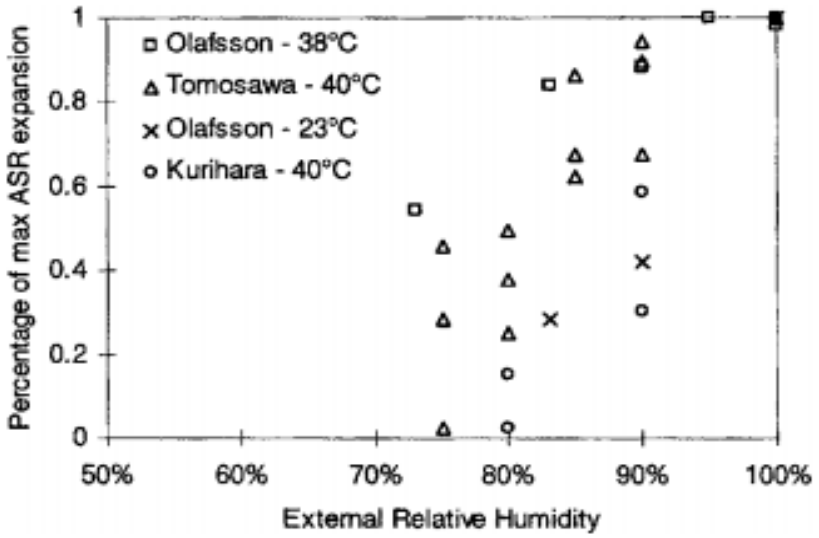


Figure 2.22. Expansion with external humidity obtained by various researchers (Poyet et al, 2006)

According to Poyet et al (2006), this might be as a result that Olafsson used a temperature of 23°C and Kurihara used a temperature of 40°C and a short duration for the test, which is too short to reach maximal ASR expansion. According to studies (Tomosawa et al, 1989; Kurihara et al, 1989; Larive et al, 2000), it has been shown that the rate of expansion due to ASR is directly proportional to the increase in mass, this might be as a result of increase in the size and weight of the silica gel, as it absorbs more moisture from its environment and gets bigger. However, the record of water ingress should be considered. (Vivian, 1981; Larvie et al., 2000). It has also been observed that the reaction will stop if internal relative humidity is less than 80% (Pedneault 1996) as shown in figure 2.23.

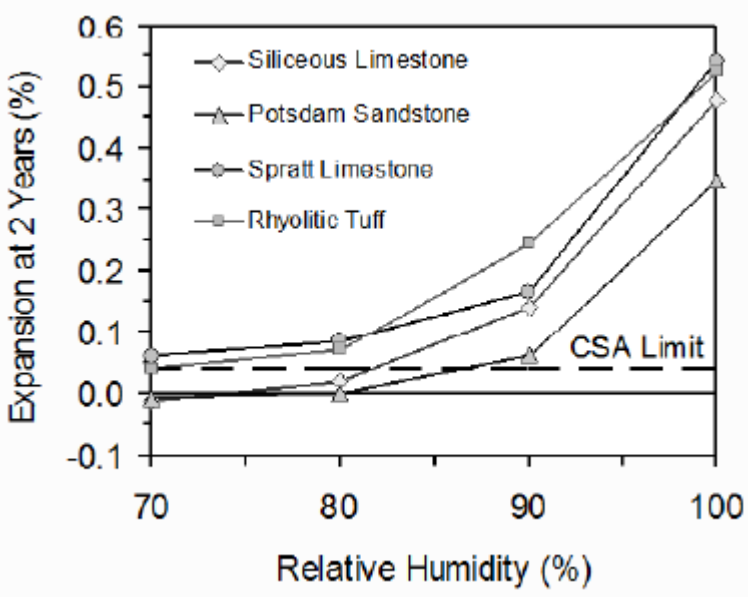


Figure 2.23 Effect of Relative humidity on expansion of concrete prism (Pedneault 1996)

Nilsson (1983) in his research concluded that when the external relative humidity is below 80% there is no alkali silica degradation which might not be true because the external humidity might be less than 80% but the internal relative humidity inside concrete could be higher which might be a good source of supply of moisture to the silica gel. According to another researcher (Ludwig, 1989) the threshold for the reaction is between relative humidity of 80% and 85% for a temperature of 20°C which is more defined as temperature affects relative humidity, therefore the value of the threshold should depend on temperature.

For Olafsson (1986), the threshold for ASR starts from about 80% relative humidity at 23°C to 75% relative humidity at 38°C. This threshold value is below 75% at 40°C for studies carried out by Kurihara et al (1989) and Tomosawa et al (1989). Based on all these studies, there is no linear relationship between the threshold relative humidity and temperature, but it should be noted that

varying the external relative humidity and temperature induces how the ionic species existing at different concentration move within the concrete (Xu et al, 1993; Rivard et al, 2003)

3 EXPERIMENTAL AND TEST PROCEDURES

3.1 GENERAL DESCRIPTION

This chapter outlines the materials, apparatus, procedures of all the experiments carried out for this thesis. Starting from selection of the appropriate sensor for measuring moisture in concrete, tests carried out to measure moisture in concrete and also to determine the effect of external relative humidity and water to cement ratio on alkali silica reaction are detailed.

Tests will be grouped into two phases based on the followed procedure and materials used. Phase 1 is composed of tests performed on concrete which included selection of the appropriate sensor to measure moisture and the measurement of internal relative humidity. Non-reactive aggregates were used in Phase 1. Phase 2 is composed of tests done on mortar, which comprised of finding the effect of external relative humidity on alkali silica reaction and the effect of water to cement ratio on progression of the alkali silica reaction.

3.2 SENSORS

Since it has been agreed in the literature that relative humidity is a good measure of the amount of moisture present in concrete, finding a proper and effective way of measuring this property will help to determine the moisture content in concrete. Different sensors exist in the market with different properties and capabilities. One of the main challenges is to find a sensor capable of measuring the internal conditions accurately while surviving the alkali environment of the concrete. Other things also considered are the ease in which data can be retrieved from sensors and the power source for the sensor.

To select the appropriate technology to use for this research, several sensors and probes were considered based on their properties, current and past uses, after which it was streamed down to three sensors and one probe: Sensirion SHT7x, iButton DS 1923 sensor, RapidRH sensor and Vaisala HM44 probe kit, respectively. All the sensors and probes were tested to see the best one that can be used in this research to measure the internal conditions inside concrete. A brief summary of the sensors and probe is as follows.

Sensirion SHT7x

Sensirion SHT7x is produced by a Swiss company specialized in development of sensors for different uses. SHT7x is a pin type of temperature and relative humidity sensor; the sensor has to be connected via wire to a data logger to measure the conditions, the data logger can also be connected to computer to record the conditions measured. It can measure temperature in the range of -40 to +125°C and relative humidity range of 0 - 100% RH.

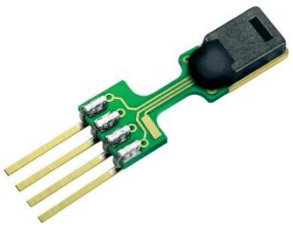


Figure 3.1 Sensirion SHT7x sensor (www.sensirion.com)

iButton DS 1923

The iButton DS 1923 sensor is being produced by Maxim Integrated and is a self-sufficient sensor that measures relative humidity, temperature and dew point and saves the result in its memory. The sensor can be configured for measurements at desired intervals and also the desired condition, i.e.

relative humidity, temperature and dew point can also be selected for measurement. The intervals of measurement vary from 1 second to 273 hours and store 8192 8-bit readings maximum; these readings can be downloaded to computer via the data logger hardware and software. The stainless-steel package makes it durable and it's able to resist environmental factors inside concrete such as moisture and dirt. One of its advantage is the ability to set the start time it starts taking measurement to a future date and time. The temperature range is -20°C to +85°C with an accuracy range of ±0.5°C in the range of -10°C to +65°C after software correction, the humidity range is 0% to 100% relative humidity. Figure 3.2 shows the sensor; the small hole allows for moisture exchange with the surrounding environment for RH measurements.

Figure 3.2 iButton DS 1293 sensor (www.maximintegrated.com)

RapidRH

RapidRH is produced by Wagner meters and mainly created to measure relative humidity in concrete floors as an alternative to the calcium chloride method. The sensor can measure both relative humidity and temperature. The temperature ranges it can measure is -20 to +110°C and relative humidity range of 0 - 100% relative humidity. To measure the conditions, the sensor has

to be inserted in the hole that the condition is to be determined, and the data viewer is connected to the sensor that shows the measured values, this sensor cannot log results as shown in figure 3.3.

Figure 3.3 RapidRH sensor (www.wagnermeters.com)

Vaisala HM44 Probe Kit

Vaisala HM44 is the probe that has been used in many published references. The probe can measure relative humidity, dew point and temperature. To measure the internal conditions at a point, the probe has to be inserted in a pre-drilled hole in the concrete. This probe is connected to a data logger that displays the real-time readings.

Figure 3.4 Vaisala HM44 Probe kit (www.vaisala.com)

Sensor comparison

A preliminary review was done on the sensors and probes available in market; the sensors properties are explained and some are summarized below and also in Table 3.1.

Size: This is the size of the sensors based on the dimensions; the size provides the ease in which these sensors can be used in measuring the internal relative humidity in concrete. As to measure concrete's internal relative humidity, hole has to be drilled in the concrete or a pre- drilled hole using a pipe, therefore to take the internal conditions, the sensors has to be able fit in into the hole drilled.

Power Source: Different sensors have different sources in which power is been supplied to it, which varies from self-sufficient sensors that have a battery to those that required connection to a data logger that supplies the energy.

Data Logging: As the conditions are being measured by the sensors, the data can be logged in various ways which can be direct logging into a computer by a software that converts the reading, or those that stores the readings in the sensors itself, or those that has a data logger which displays the recorded data at a specific point in time or in a continuous manner depending on its configuration.

Data Collection: After the sensors have collected all the data, one of the critical things is the ease in which this recorded or displayed data can be collected so as to analyzed, with the evolution of

technology, the nominal way of writing out data displayed by data loggers has been improved as some of the data's logged are saved in excel format and the file can easily be copied from a computer and analyzed, also for the energy self-sufficient sensors, these sensors has just to be connected to an interface and the data logged can also be harvested in an excel sheet format.

Connection type: Based on the energy supplied to the sensors, the way the sensors are connected to measure condition to logging data varies from wired to non-wired, i.e. some require the sensor to be connected via a cord to the data logger.

Cost: This is the financial value of these sensors, though some of these sensors are really small in size, the price can range from a hundred dollars to two thousand dollars, so when using these sensors, the cost as to be considered so as to have a cost-effective process.

Criteria	Sensors			
	Sensirion SHT7x	**iButton DS 1293**	**Vaisala**	**RapidRH**
Size of Sensor	19.5 x 5.08 x 3.1 mm	18 mm dia. x 6mm	Probe =12 mm dia x 69 mm Cable length = 0.3 m	Diameter = 19mm Length = 120mm
Humidity Range **Accuracy** **Sensitivity**	0-100% n/a 0.01%	0-100% (±5%) 0.1%	0-100% (±2 %) 0.1%	0-100% n/a 1%
Temp. Range **Accuracy** **Sensitivity**	-40 to +125°C - 0.01°C	-20°C to +85°C (±0.5°C) 0.1°C	-40 to +60 °C (±0.4 °C) 0.1°C	-20 to 110°C - 1°C
Suitable Installation Method	Embedded in fresh concrete and hardened concrete	Embedded in fresh concrete and in hardened concrete	Embedded in fresh and hardened concrete	Hardened concrete is drilled
Data logging	By a data logger	By the sensor itself	By a data logger	No logging
Data Collection	Using an easy-to use plug-and-play System Evaluation Kit EK-H4	Readings are stored in the memory system within the iButton	A data logger is connecter with the probe to the sensor	Has a visual display
Energy Source	From the data logger	Self sufficient (internal battery)	From the data logger	From the reader
Accessories	Evaluation kit EK-H4	-iButton connectivity kit -iButton Retainer	-------	-drill -sleeves
Price (USD)	660.34 (Sensors and data logger)	141.61(sensor)	839.00 (Probe and data logger)	310.00 (Sensors, sleeves and data viewer)

Table 3.1 Sensor types with their properties

3.3 MATERIALS

3.3.1 PHASE 1

For the first phase of the experiments, concrete was made and the internal relative humidity inside the concrete samples was measured. For the first stage of phase 1, the concrete consists of coarse aggregate, fine aggregate, cement, fly ash (in some cases) and water. The mixture designs used are shown in Table 3.2. Limestone coarse aggregate with a maximum size of 10mm and a natural sand were used. It was observed that dirty materials coated the coarse aggregate, so it was washed and dried as shown in figure 3.5. The aggregates were dried at room conditions for approximately 24 hours before using them to make the concrete. While all the samples used for experiment 1a are composed of concrete without SCMs or coatings, only three samples out of the nine samples used for experiment 1b is composed of only basic concrete components (cement, water and aggregates), three other samples are composed of 50% fly ash to see the effect the fly ash has on the internal relative humidity inside concrete and the remaining three samples were coated with epoxy to observe the effect on internal relative humidity inside concrete.

Material	Portland Cement Mixture (kg)	Fly ash Mixture (kg)
Fine aggregate	4.12	4.12
Coarse aggregate	6.18	6.18
Cement	2.06	1.03
Fly ash	0.00	1.03
Water	1.03	1.03
Total	13.29	13.29

Table 3.2 Material quantities for Phase 1

Figure 3.5 Washed coarse aggregate

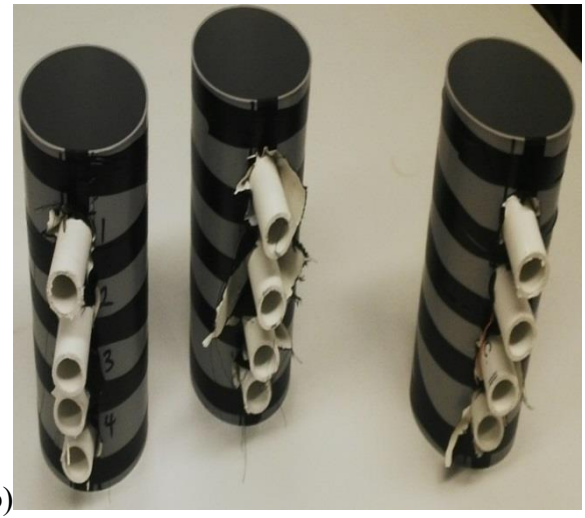
3.3.2 PHASE 2

This phase of experiments is composed of determining the effect of external relative humidity and water to cement ratio on alkali silica reaction. In this phase, mortar samples were used. The same Portland cement for Phase 1 was used. The aggregates used were Spratt fine reactive aggregates that have been proven to be ASR reactive both in field and lab studies. The aggregate was crushed to obtain the required aggregate sizes and was graded according to the requirement for ASTM C 1260 test procedure. The mixture proportion for this test was 1 part of cement to 2.25 parts of the graded aggregate. For stage 2a, the water cement ratio remained constant at 0.47 as per the standard. For stage 2b, water cement ratios of 0.45, 0.47, 0.50, 0.55 and 0.60 were investigated. The mortar was mixed in accordance with requirements for ASTM C 305. For the second phase of experiments, all the samples used are of dimension 50mm x 50mm x 50mm using the standard mold for making cubes.

3.4 TESTING PROCEDURES

3.4.1 PHASE 1

For the first phase, all the samples used are cylindrical with height of 200 mm and diameter of 100 mm. The molds used to cast these samples were modified by drilling holes in the mold so as to allow the sensors to be inserted to measure the internal relative humidity in concrete; this is shown in figure 3.6.

(a)  (b) 

(c) 

Figure 3.6 (a) the holes bored in the molds, b) the side of tube covered by Gortex was inserted in the mold, c) The other end of the tube was covered by a rubber stopper

Plastic tubes were inserted in the holes to specified depths to allow for moisture measurements. The end of the tubes inserted in the mold was covered by Gortex material to avoid concrete coming into the tube during casting, the other end of the tube was blocked by a rubber stopper to avoid the loss of relative humidity and ensure that an accurate measurement is obtained. The height and depth of the tubes varied and will be explained in a later section.

3.4.1.1 Investigation of Sensors (Experiment 1a)

The concrete was cast and cured for approximately 24 hours in the mold, including a top cover, after casting. After this time, the outer cylinder mold was removed and the sensors were inserted into the plastic tubes. The position of the sensor in each sample is described in Table 3.3 and figure 3.7 shows the sensor location schematics.

Sensor	Placement of Sensor	
	Height of sensor (mm)	Depth of Gortex (mm)
iButton	30	15
Vaisala	70	15
Sensirion	110	15
RapidRH	150	15

Table 3.3 Position of the sensors

NB: The height of sensor is with reference to the base of the sample and the depth of Gortex is with reference to the side of the sample.

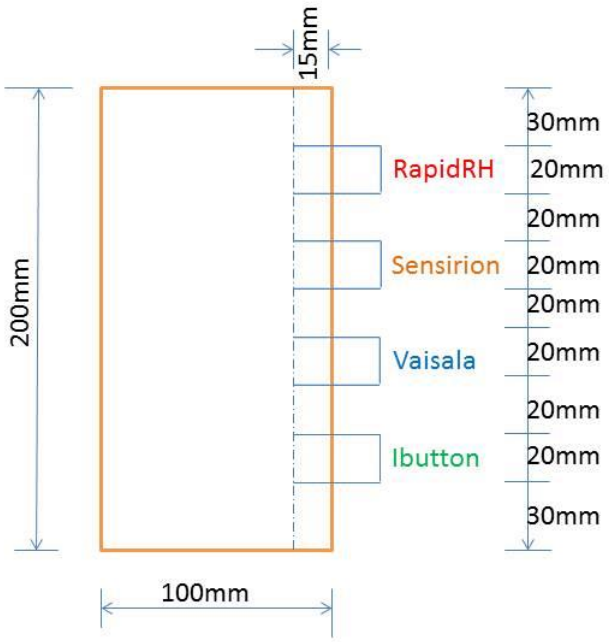


Figure 3.7 Location of sensors in the mold – Phase 1a

The environmental condition for this experiment is 100% relative humidity and 38 degrees Celsius similar to ASTM C 1293. To obtain this relative humidity, improvisation was done by using distilled water in an enclosed container. It was noticed that the tap water used couldn't produce 100% relative humidity, so distilled water was used in subsequent experiments. The enclosed container with the concrete samples and sensors inserted was placed in the oven and the temperature was set to 38 degrees Celsius.

(a)  (b) 

(c) 

(d)  (e) 

Figure 3.8 (a) iButton sensor attached to a rubber stopper, b) curing of the concrete samples after casting, c) concrete sample after removal of mold after 24 hours curing, d) container in which samples are placed to regulate the relative humidity, e) the whole setup place in the oven.

Three replicate samples were tested for this phase. As there was only one Vaisala probe available and to obtain a reliable internal moisture evolution and accurate results, the Vaisala probe was placed in only one of the three samples. All other sensors are inserted into the plastic tube in each of the sample and for the iButton sensor; it was attached to the rubber stopper as shown in figure 3.8.

The Sensirion sensor being a wired sensor was connected to the computer for the duration of the experiment for continuous monitoring and logging of data. To take measurement from the RapidRH sensor, the rubber stopper had to be removed and the reader connected with the sensor to take the measurement. The Vaisala probe was connected to the data logger and readings were taken at 24 hours intervals. The iButton sensor is also activated an it is configured to take the relative humidity and temperature readings at 24 hour intervals. Measurements were taken for 28 days.

The sensors were also tested in a controlled in a controlled environment to see the variation in readings of relative humidity in similar sensors. This was done by putting all the sensor in a confined box with approximately 75% relative humidity and measuring the relative humidity at all sensors at different times during the day. Sodium chloride was used to control the relative humidity.

3.4.1.2 Depth of Sensors (Experiment 1b)

After casting and 24 hours curing, the cylinder mold was removed and all the samples were coated at the top and bottom sides of the cylinder with epoxy to prevent loss of moisture; the only loss of moisture was from the cylindrical surface. Humidity measurements were taken at 3 points in all these samples and tagged point 1, point 2 and point 3. The specifications of the points are as follows and showed in the schematic in figure 3.9.

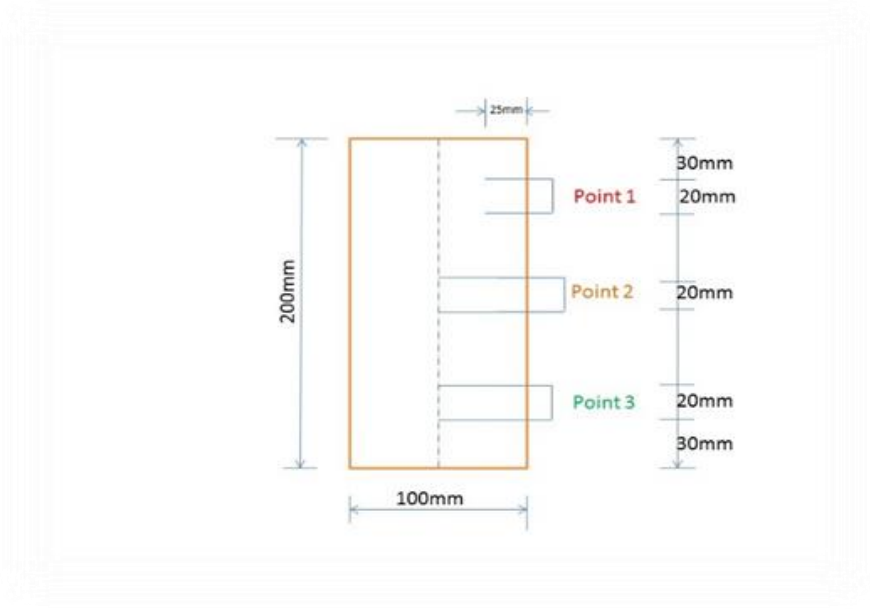


Figure 3.9 Position of sensors in the sample – Phase 1b

- Point 1; 30mm from the top of sample and 25mm into the concrete
- Point 2; 90mm from the top of the sample and 50mm into the concrete
- Point 3; 30mm from the base of the sample and 50mm into the concrete.

Point 2 is at approximately at the centre of the sample in both depth and distance from surface and since both ends of the concrete samples are coated, point 1 and point 3 have the same properties since they both have equal distance from the end of the sample, the difference in the relative humidity measurements at point 1 and 3 would be as a result of the different depths i.e. point 1 is

25mm into the concrete while point 3 is 50mm into the concrete. Only iButton sensors were used for this part of the experiment, the samples are placed at a fixed temperature but varying relative humidity conditions starting with 100% relative humidity, then 75%, 65% relative humidity and back to a higher relative humidity of 95%. The samples are exposed for 14 days for each of the relative humidity conditions.

3.4.2 PHASE 2

In this phase, the standard ASTM C 1260 was followed, the only alteration done was the use of cube mortar samples instead of mortar bars. For both experiments 2a and 2b, the conditioning was the same for the first 14 days as follows:

Conditioning

- The temperature of the lab when the samples were cast was between 22°C and 24°C
- The mixing water temperature was 21.7°C
- The relative humidity of the lab when the samples were cast was 55%
- The storage oven in which the samples were stored was 80.0°C and constant measurement of temperature was done to ensure that this does not vary more than ±2.0°C

3.4.2.1 Experiment 2a

In this experiment, 15 mortar cube samples were cast using the same water to cement ratio of 0.47. The samples follow standard procedure of ASTM C 1260, the only exception was the use of cube mold of 50mm x 50mm x 50mm which allows measurement of length changes on all sides of the sample. Each mold was covered immediately after casting to prevent loss of moisture and remain

in the mold for approximately 24 hours. The samples are removed after 24 hours and labelled while preventing loss of moisture.

The samples were then placed in plastic containers with sufficient tap water to ensure that they are all fully immersed. The container was sealed and placed in the oven for approximately 24 hours at a temperature of 80°C. After storing the samples in water at 80°C for 24 hours, the containers were removed from the oven and the cubes removed one at a time to take the zero readings after drying with a towel to the nearest 0.0005mm using a digital Vernier caliper. Each face was measured at three pre-marked locations and the expansion was determined based on the average percentage change in the surface area of each sample. Figure 3.10 shows the schematics for the locations of the lines on the mortar samples in which the length is measured. Figure 3.11 shows a typical view of how lines were drawn on each side. Since the surface shape is a square; the surface area for the sample was calculated by multiplying the average of length measurements on each side (i.e. average of 18 length measurements obtained from 3 length measurements on each side of the sample) multiplied by the average width (i.e. average of 18 width measurements obtained from 3 width measurements on each side).

After the zero readings of all the samples have been taken, they were placed into another container and filled with 1N NaOH in such a way that all the samples are fully immersed in the sodium hydroxide solution. The container was then sealed and placed in the oven at a temperature of 80°C for 14 days to allow for expansion of the sample caused by ASR.

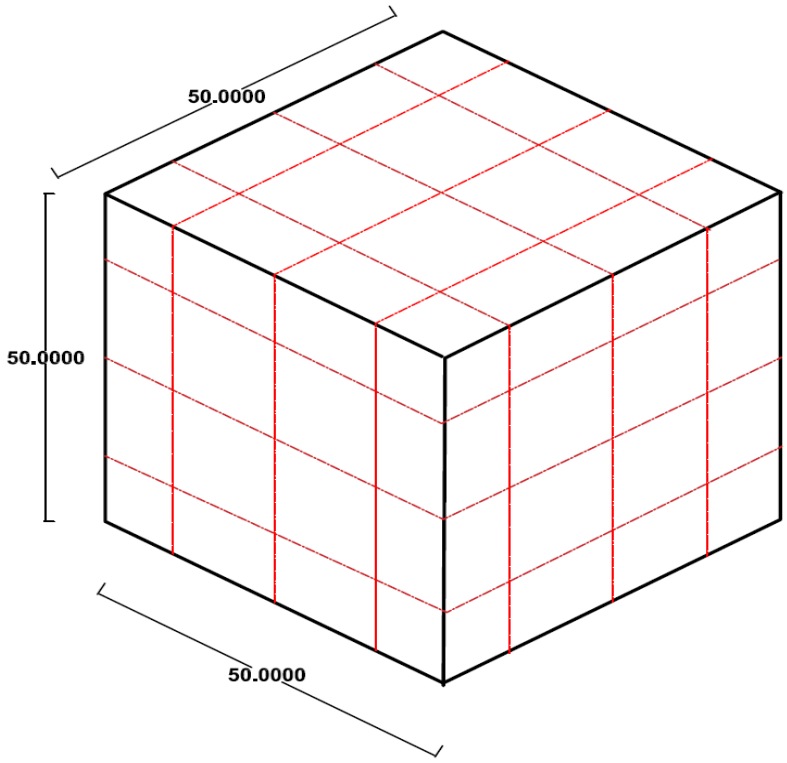


Figure 3.10 Schematics show the lines on which measurements are taken

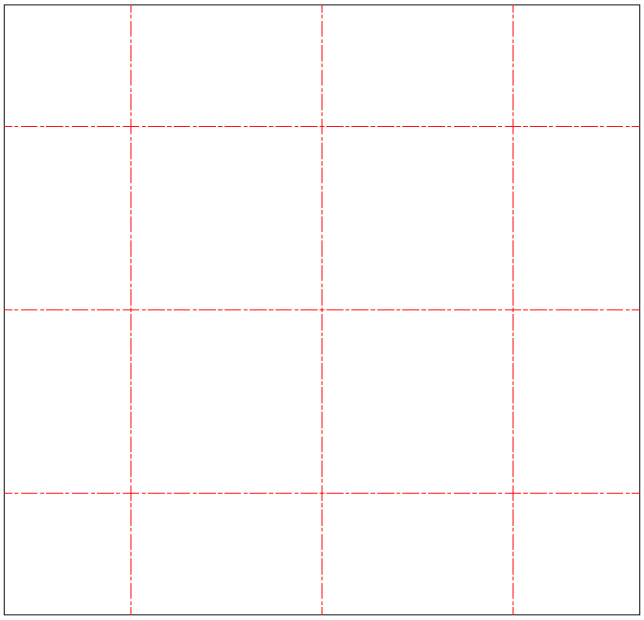


Figure 3.11. Typical view of each side of the mortar sample

After the samples were stored in sodium hydroxide solution for 14 days, they were removed from the solution and Day 1 reading was taken. The samples are then exposed to 4 different relative humidity conditions and measurements also taken at 4 days, 7 days, 14 days and 28 days after removal from the sodium hydroxide solution. The salts used to control the relative humidity at 38˚C are (75% expected relative humidity), sodium nitrate (63% expected relative humidity), Magnesium Nitrate (48% expected relative humidity) and magnesium chloride (33% relative humidity). Water was used to obtain a relative humidity of 100% and saturated salts are used to obtain other relative humidity as shown on Table 3.4 and placed inside oven at 38˚C.

Salts	**Expected Relative Humidity (%)**	**Average Relative Humidity Measured**
Sodium Chloride	75%	75%
Sodium Nitrate	63%	60%
Magnesium Nitrate	48%	46%
Magnesium Chloride	33%	33%
Room	n/a	35%

Table 3.4 Salts used in controlling Relative Humidity

For ease, the relative humidity in the results are referred to as 100%, 75%, 60%, 45% and room respectively. Measurement of all the sides of each cube sample was taken at the stated days and the surface area of each cube calculated and recorded. The difference between the Day 1 surface area readings surface area each other day was calculated. The expansion for this experiment is

expressed as the average percentage change in surface area of samples in the same exposure conditions.

3.4.2.2 Experiment 2b

In this experiment, different five different water to cement ratios (0.45,0.47, 0.50, 0.55 and 0.60) were tested. Each batch consisted of 3 cubes and were cast, cured and placed in water followed by 1M NaOH as in Experiment 2a. The samples were left in the 1M NaOH solution for 14 days and the change in the surface area of all the sides of the samples are taken using a digital Vernier caliper to take the length of each side of each sample. The change in surface area of each samples was calculated, and the average for change in surface area of samples with the same water to cement ratio calculated.

4 RESULTS AND DISCUSSION

4.1 PHASE 1

4.1.1 EXPERIMENT 1A

To test the repeatability of the sensors, several sensors of each type (except for the Vaisala) were placed in a glovebox controlled to 75% RH with the use of saturated NaCl. Table 4.1 shows the measurements obtained over 3 days. It can be seen that except for I2, each sensor had nearly constant and stable readings. The Vaisala sensor measured the relative humidity consistently less than the hygrometer in the glove box (BH); the others were consistently higher. All sensors were placed approximately 15 cm above the tray of saturated salt; while the box hygrometer was near the top of the glovebox. It is possible that with the addition of a fan; the sensors would be closer to that of the glovebox. For the Vaisala sensor; it is possible that it was not recently calibrated. It can also be seen that within the iButton and Sensirion sensor types, the RH measurements were generally within 2% RH.

Day	Time	BH	R1	R2	R3	V	I1	I2	I3	S1	S2	S3
1	a	72	76	73	77	70.70	79.0	80.3	79.0	77.06	75.89	74.88
1	b	72	76	73	77	70.70	79.0	80.3	79.0	76.94	75.92	74.69
1	c	72	76	73	77	70.70	79.0	79.2	79.0	76.88	75.97	74.66
2	a	72	76	73	77	70.70	79.0	81.0	80.0	76.85	75.81	74.63
2	b	72	76	73	77	70.70	78.5	81.0	80.0	76.85	75.81	74.62
2	c	72	76	73	77	70.70	79.0	81.0	80.0	76.85	76.10	74.71
3	a	72	76	73	77	70.80	79.0	81.0	80.0	77.03	76.49	74.87
3	b	73	76	73	77	70.70	79.0	81.3	80.0	77.21	76.57	75.05
3	c	73	76	73	77	70.80	79.0	81.3	80.0	77.52	75.61	75.21

Table 4.1 Repeatability Measurement of Sensors

From table 4.1; BH = box hygrometer, R = RapidRH sensor, V = Vaisala, I = iButton sensor, S = Sensirion sensor. The number in front of the letter is just for identification.

The concrete samples exposed to 100% relative humidity and 38 degrees Celsius were monitored for 28 days after curing. The sensors used are referred to as RapidRH, Sensirion, Vaisala and iButton. As noted in Chapter 3, the Vaisala probe was only used in sample 2. Further details about the sensors and the location are shown in figure 3.7 and Table 3.3.

The following results were obtained for the duration of 28 days that the samples were exposed to the environment. Figures 4.1 to 4.3 show the temperature trend observed for each sample and it can be noted that the variation at any time is within approximately 2ºC between the sensors for the entire 28 days and for all the sensors for samples 1, 2, and 3, respectively. It should be noted that the RapidRH sensor has the lowest sensitivity and measures to the nearest 1°C. The temperature of the oven was taken once every day and the average for the duration of the test was 38˚C. In order to measure the temperature using the RapidRH sensor and the Vaisaila probe; the oven had

to be opened and the RapidRH reader and Vaisala probe inserted and left for approximately 5 minutes before the readings are taken while for the Sensirion sensors, the temperature was logged every 20 minutes and every 24 hours for the iButton sensor.

It can be observed from figures 4.1 to 4.3, that the result obtained from the Sensirion sensors has low variations as the results plotted for each day is the average of all the results recorded for each day (~72 readings). There was a technical glitch with the oven used which resulted in lower temperature on the fourth day and was noticed as the oven's temperature recorded for the fourth day was 36˚C instead of the set 38˚C. It was also observed from figures 4.1 to 4.3 that none of the samples reached the set temperature of the oven throughout the duration of the test.

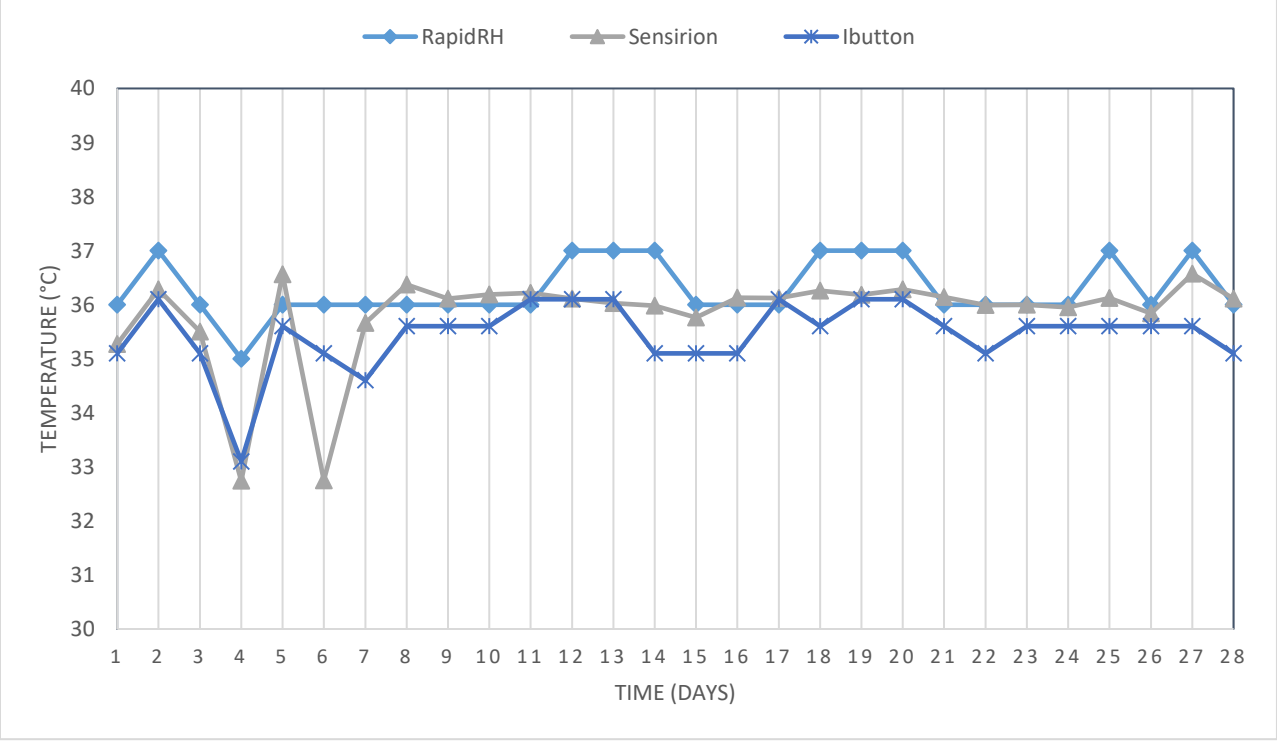


Figure 4.1 Phase 1a Temperature results for sample 1 at 38˚C and 100% RH

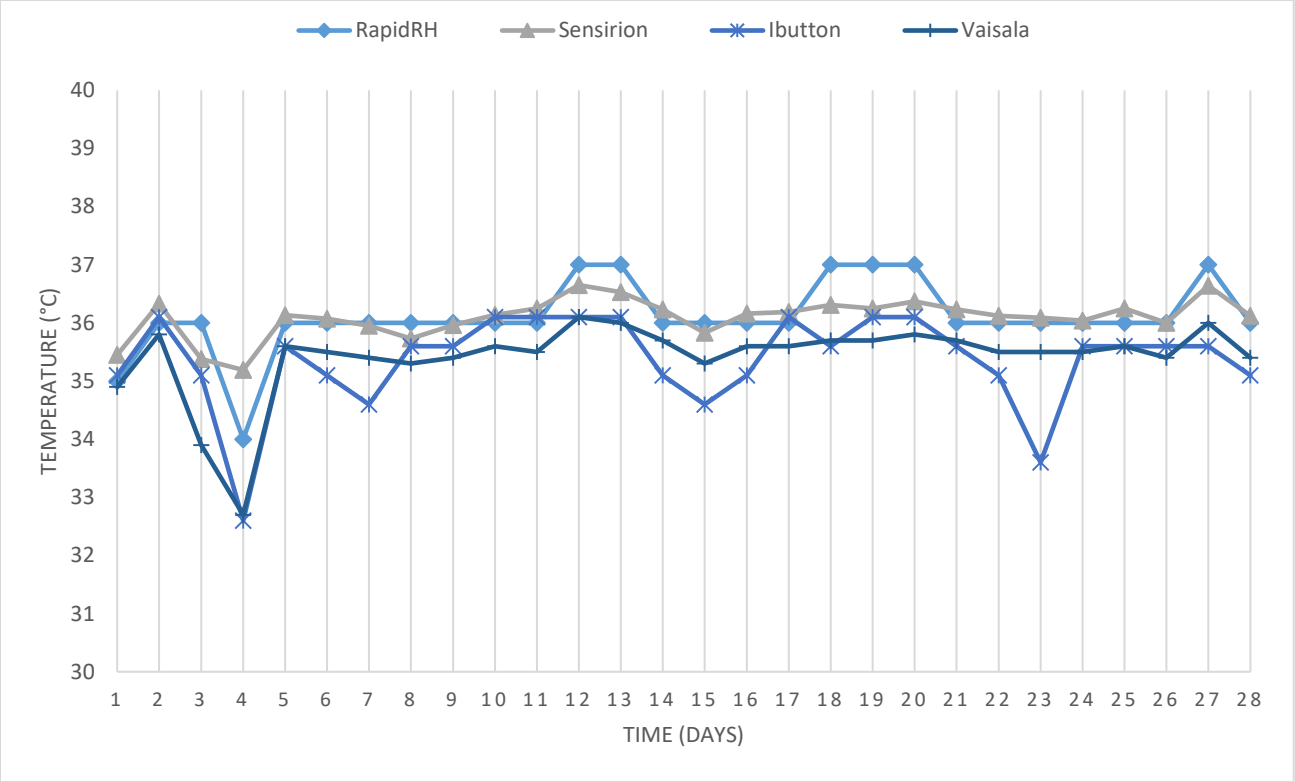


Figure 4.2 Phase 1a Temperature results for sample 2 for all sensors at 38˚C and 100%RH

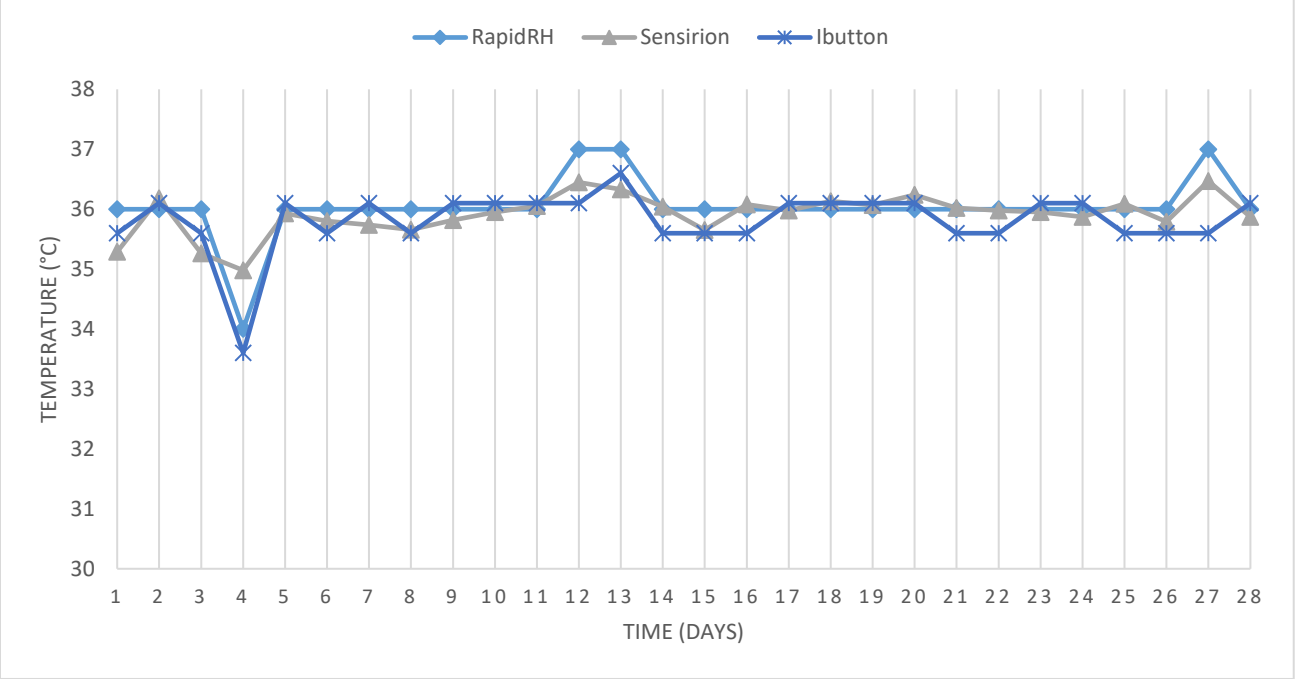


Figure 4.3 Phase 1a Temperature results for sample 3 for all sensors at 38˚C and 100%RH

The relative humidity results are presented in figures 4.4 to 4.6. The relative humidity for this study was controlled using water with an expected relative humidity of 100%, it can be observed from figures 4.4 to 4.6 that there was a sharp drop in the relative humidity for all the samples and sensors on the 13th day; this is as a result of loss of water from the container; the water was refilled on the 13th day when this was noticed.

Although all the sensors are placed to measure internal relative humidity at the same depth from the side of the samples, the difference in the relative humidity measured for each type of sensor is as a result of different position of the sensors in the samples from the base of the sample as it was stated on Table 3.3 and can be seen on figure 3.7 with RapidRH sensor being closer to the top followed by Sensirion, Vaisala and iButton sensors respectively. Also, it was noticed that tap water couldn't produce a relative humidity of 100%; the relative humidity produced ranged between 78% to 84%, this was corrected in phase 1b experiment by using distilled water to control the relative humidity as this type of water does not contain impurities.

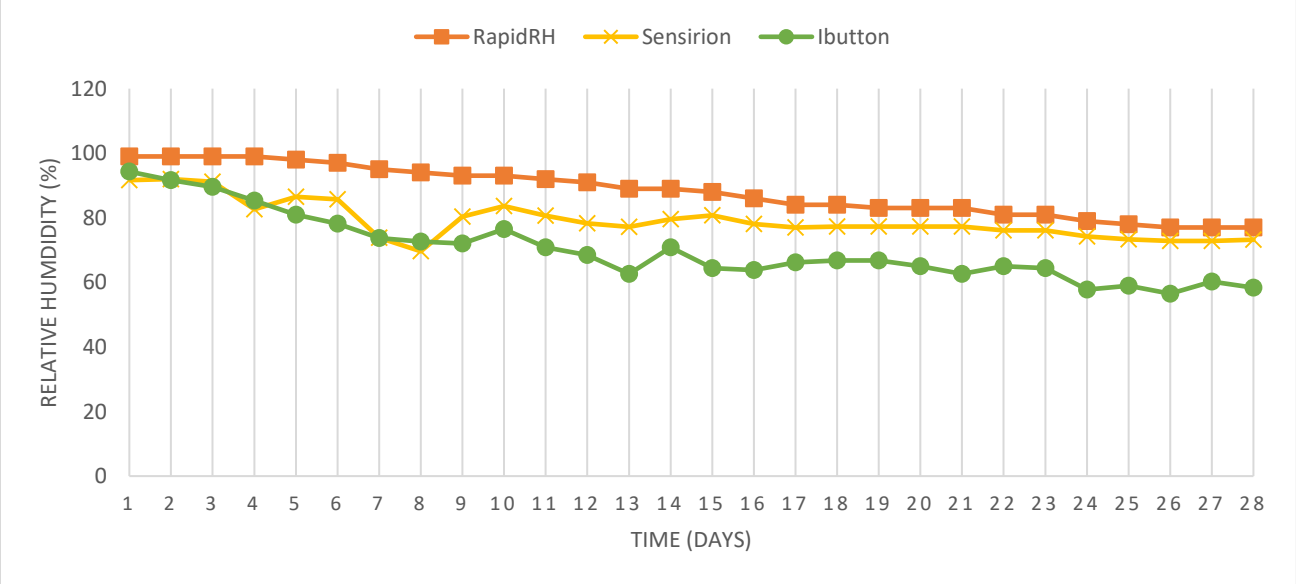


Figure 4.4 Phase 1 a Relative humidity results for sample 1 at 38˚C and 100% RH

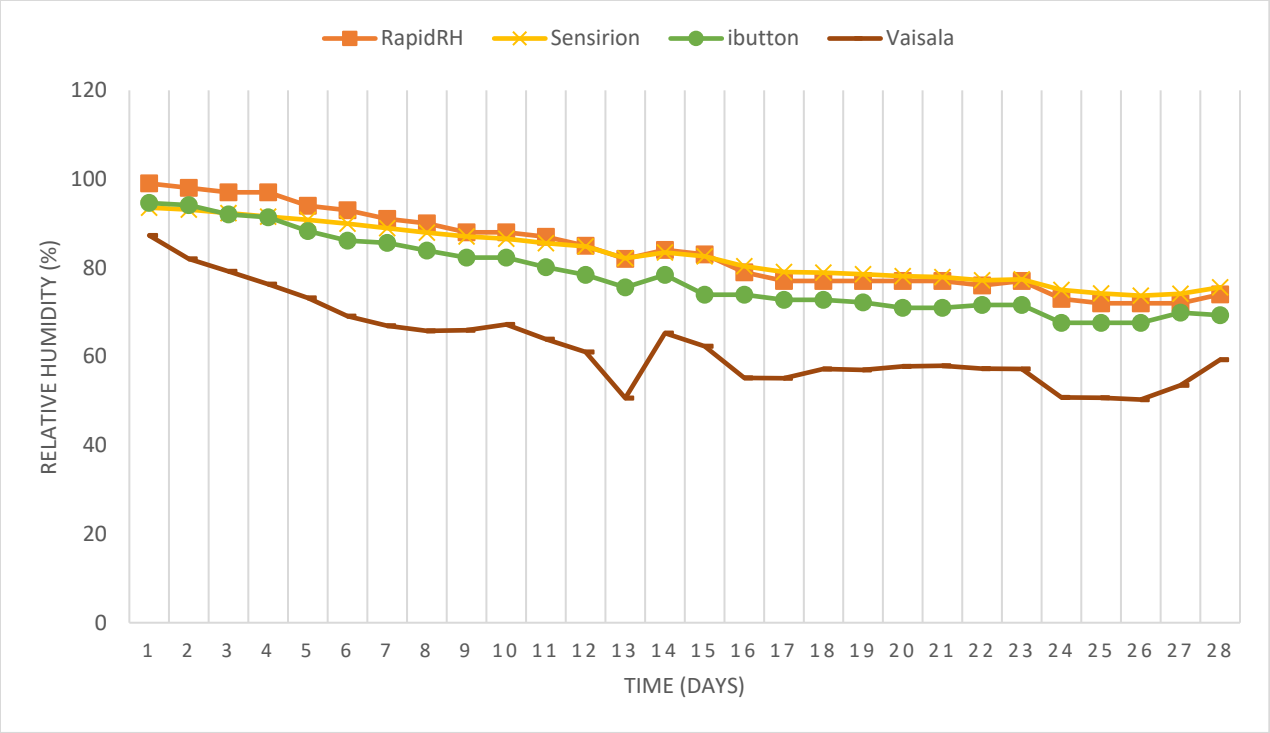


Figure 4.5 Phase 1a Relative humidity results for sample 2 at 38˚C and 100%RH

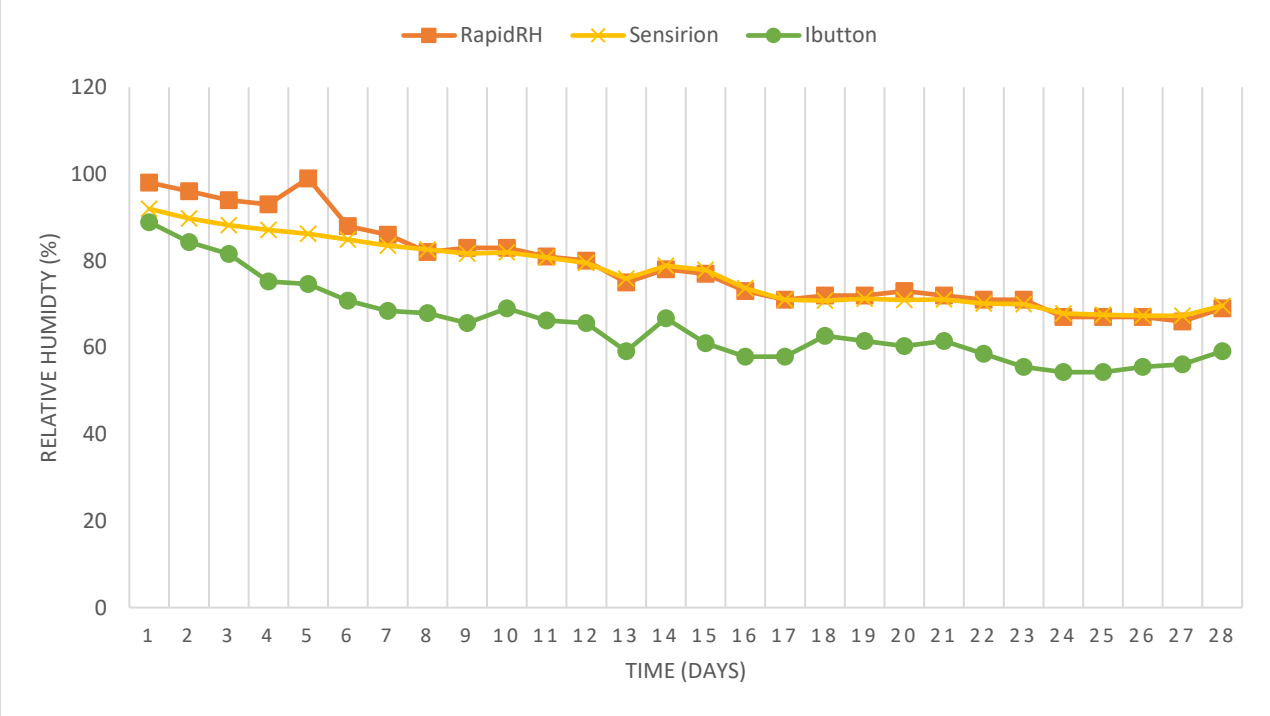


Figure 4.6 Phase 1a Relative humidity results for sample 3 at 38˚C and 100%RH

In this experiment, the outstanding sensors are the Sensirion and iButton sensors. The Sensirion was one of the outstanding because it does not require removal and insertion of sensors to take measurements; the sensors are placed in the samples permanently and the data are logged on the computer and the result correlates to two of the other sensors. However, it does require a continuous connection to record data, which might limit its usefulness in the field. Also, iButton sensor was outstanding because it can be used without removing it from the sample when taking the measurements, the sensor is placed permanently and brought out subsequently when results are needed to be harvested. The disadvantage is that there is a risk that the iButton sensor might stop working, and this cannot be discovered unless the sensor is connected to the computer. While for Sensirion sensor, if any of the sensors stopped working, it will be observed immediately on the data logger and computer and this can be replaced immediately as this happened on the 9^{th} day of Phase 1a experiment when the Sensirion in sample 1 stopped working and had to be replaced.

Based on overall assessment of the sensors tried, the sensors are rated in terms of being ideal of measuring relative humidity in concrete as follows: iButton (Best), Sensirion (Good), Vaisala (OK), RapidRH (Fair).

The iButton sensors being ranked the best was used for further experiments carried out as this has proven to be an ideal sensor which is capable of measuring the internal conditions accurately, withstand the alkaline environment, have easy access to data measured and also economical in terms of cost.

4.1.2 EXPERIMENT 1B

As the aim of this part of the experiment was to observe the effect of SCM and coating on the internal relative humidity in concrete. In this part, the measurement of internal relative humidity inside concrete was acquired using the iButton sensor. This sensor was selected for its sensitivity, small size and self-contained data logging capabilities. The samples were subjected to various external relative humidity and one fixed temperature to see the trend that occurs for the moisture in concrete based on the internal relative humidity. The samples were first subjected to 100% relative humidity, followed by 75% and 65%, respectively and later to a higher relative humidity of 95%. For the 100% relative humidity, distilled water was used to control the condition and the external relative humidity was measured alongside the internal relative humidity to see that the desired 100% relative humidity was obtained.

The following results were obtained when the sample is exposed to 100% relative humidity at room temperature after curing for approximately 24 hours. Figures 4.7 to 4.9 shows the results obtained for each sample when exposed to 100% relative humidity and a temperature range of 23°C to 25°C. The results consist of the internal relative humidity measured at each of the three points and also in all the 9 samples with 3 samples having the same properties; i.e. 3 samples with 50% fly ash (A, B, and C), 3 samples with coating (G, H and I) and 3 samples without coating or fly ash (D, E, and F.)

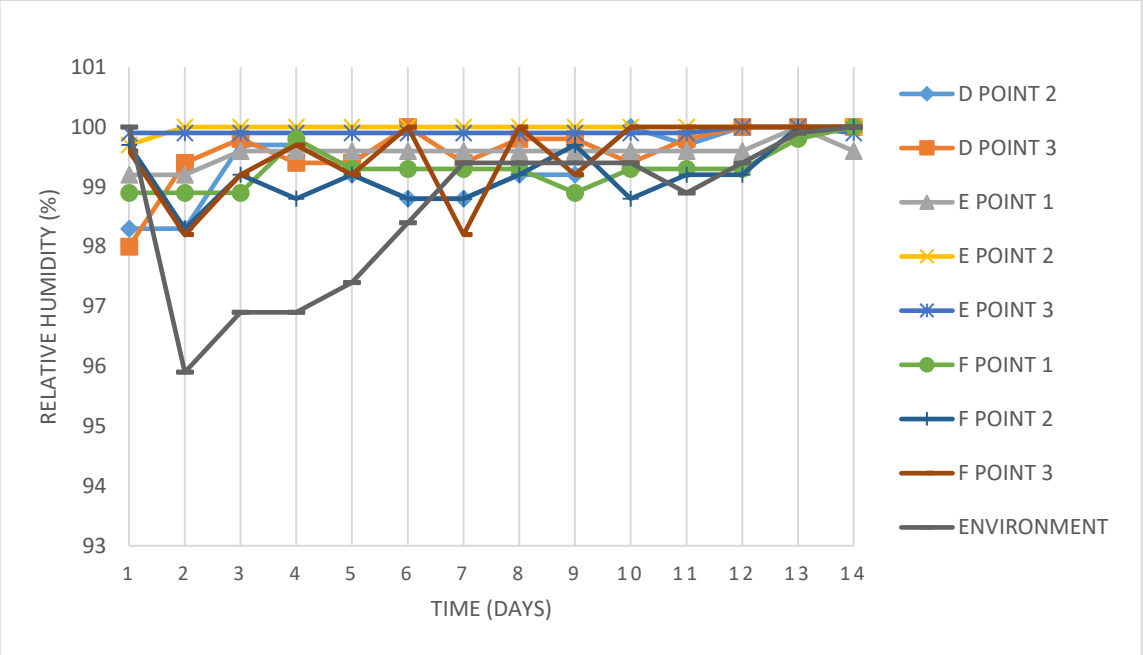


Figure 4.7 Internal relative humidity when exposed to 100% relative humidity in samples without SCM

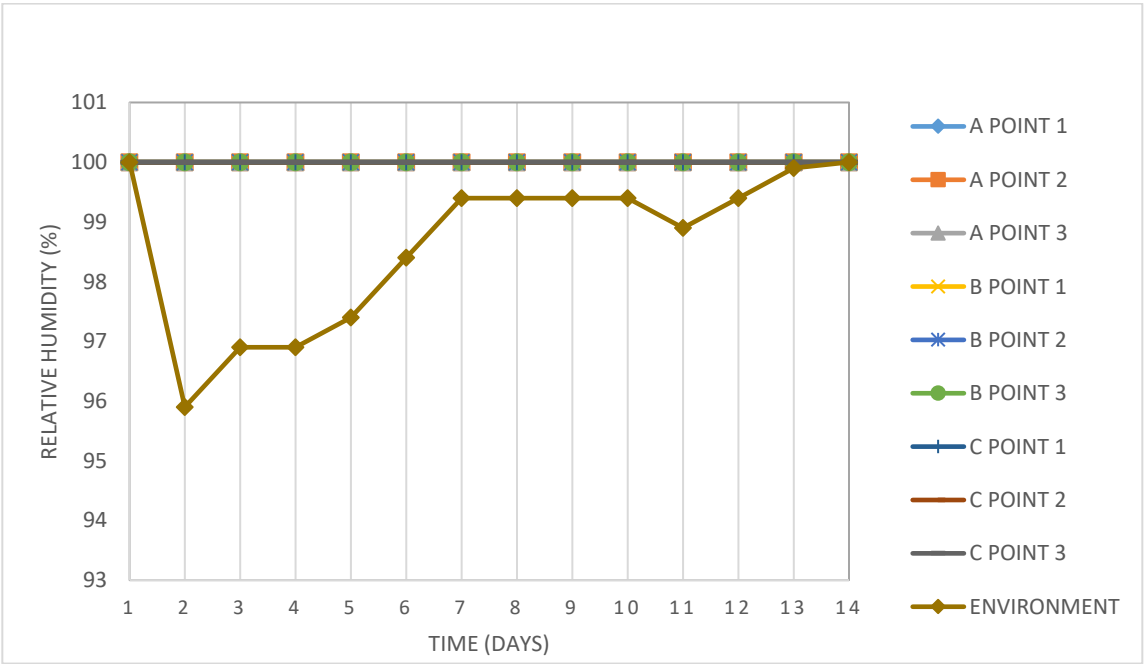


Figure 4.8 Internal relative humidity when exposed to 100% relative humidity in samples with 50% fly ash

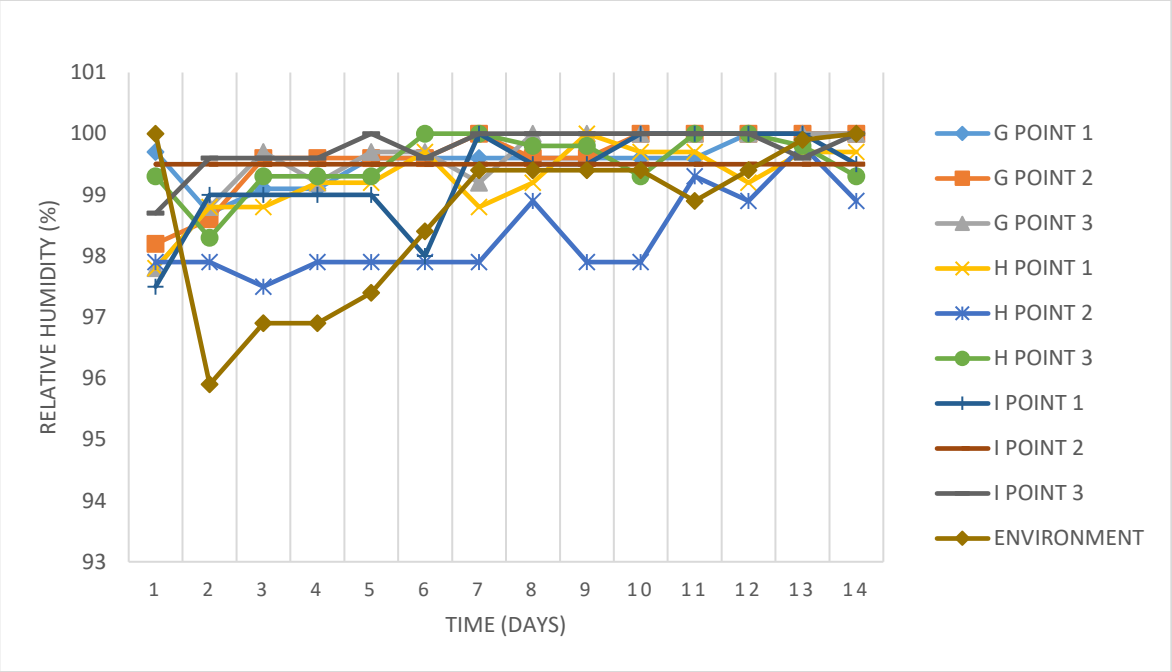


Figure 4.9 Internal relative humidity when exposed to 100% relative humidity in samples with coating

It was observed that despite the water used to control relative humidity at 100%, the measured humidity in the container holding the samples falls between the 2nd day and 7th day within 96% to 99% (Figures 4.7, 4.8 and 4.9). However, there was not a significant drop in the internal relative humidity in all the samples during these 5 days.

From Figure 4.7 which shows the results obtained for samples with 50% fly ash, it was observed that the internal relative humidity in these samples at all times was 100% on the average for throughout the duration of test while for other samples without fly ash or coating (Figure 4.8) and samples with coating (Figure 4.9) does not have the same internal relative humidity for all days. The minimum reached for all the samples placed in this condition was 98.5%; the results were further elaborated as shown in figures 4.10 to 4.12 which shows the overall average obtained for each mixture at each point, i.e. for example figure 4.10 shows the average measured internal

relative humidity at point 1 only and figure 4.13 shows the average measured relative humidity at all points. In all cases, the fly ash mixture without coating had internal relative humidity the highest at a consistent 100%. The fly ash with the coating generally had the lowest measured humidity. For this case, external humidity was not able to penetrate the coating and therefore the internal relative humidity was lower than the other two sample sets. For the Portland cement mixture, cement hydration would start to consume the mixing water at a higher rate, perhaps lowering the internal humidity as well. To account for uncertainty in the averages calculated, standard deviation error of the average is shown on some figures using error bars to show the dependency of the average calculated on the sample size.

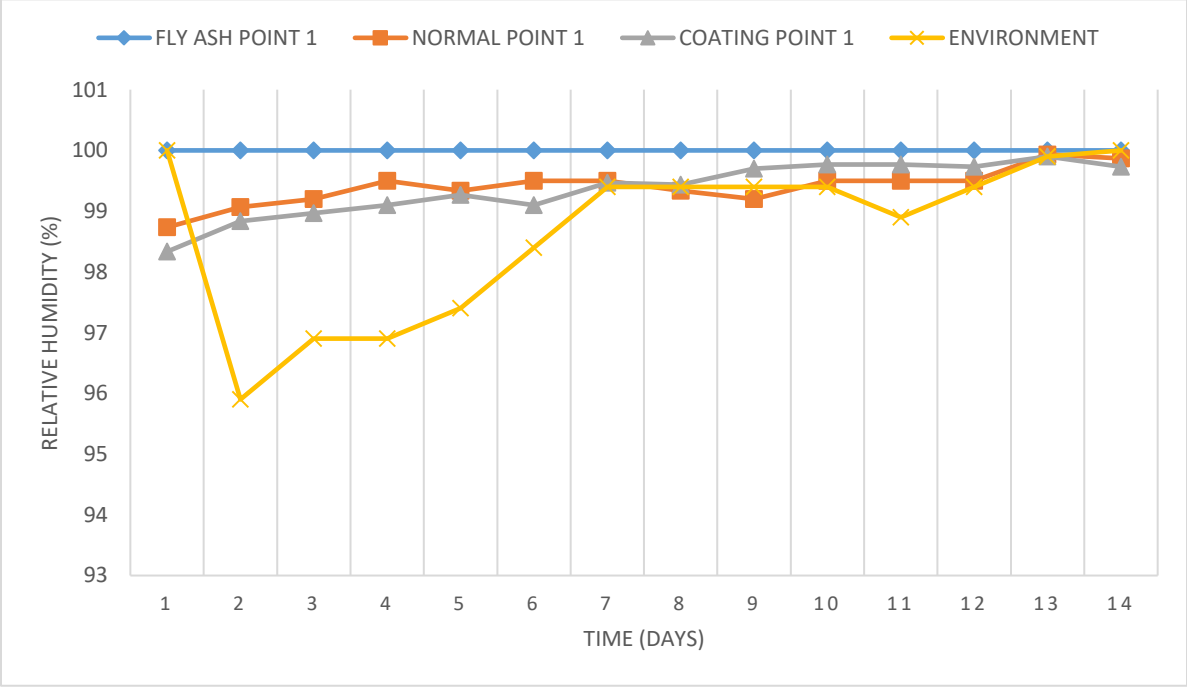


Figure 4.10 Internal relative humidity at Point 1 when samples were exposed to 100% relative humidity

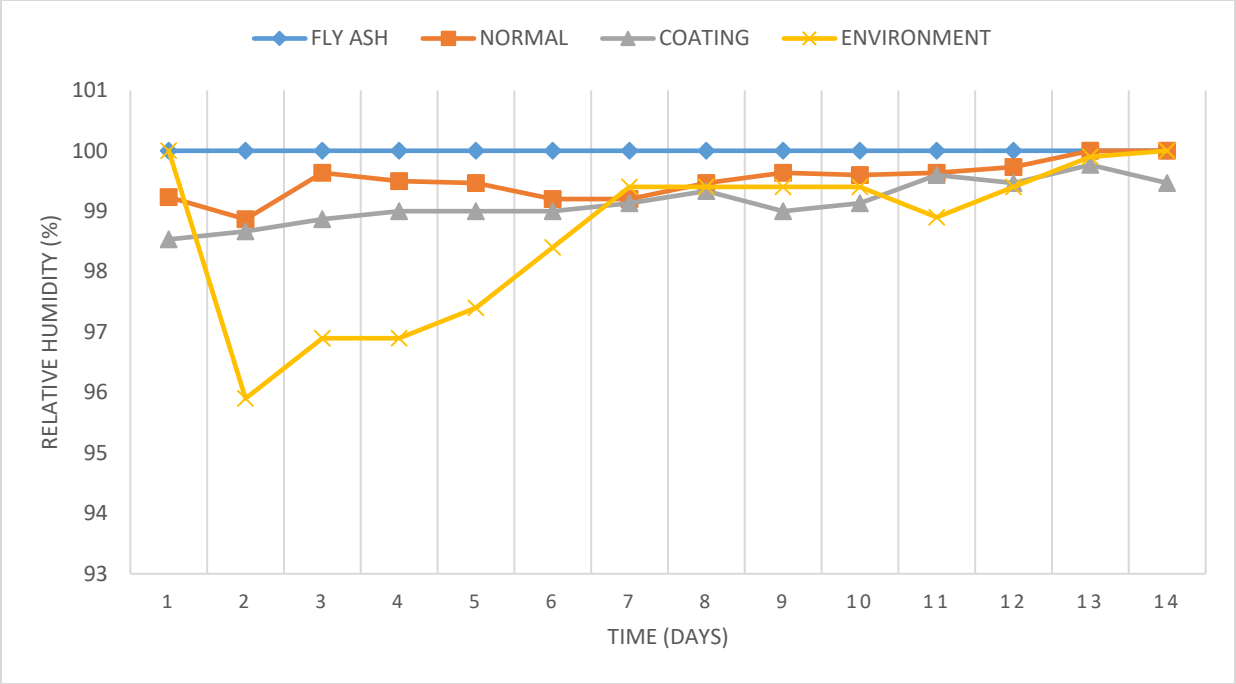


Figure 4.11 Internal relative humidity at Point 2 when samples were exposed to 100% relative humidity

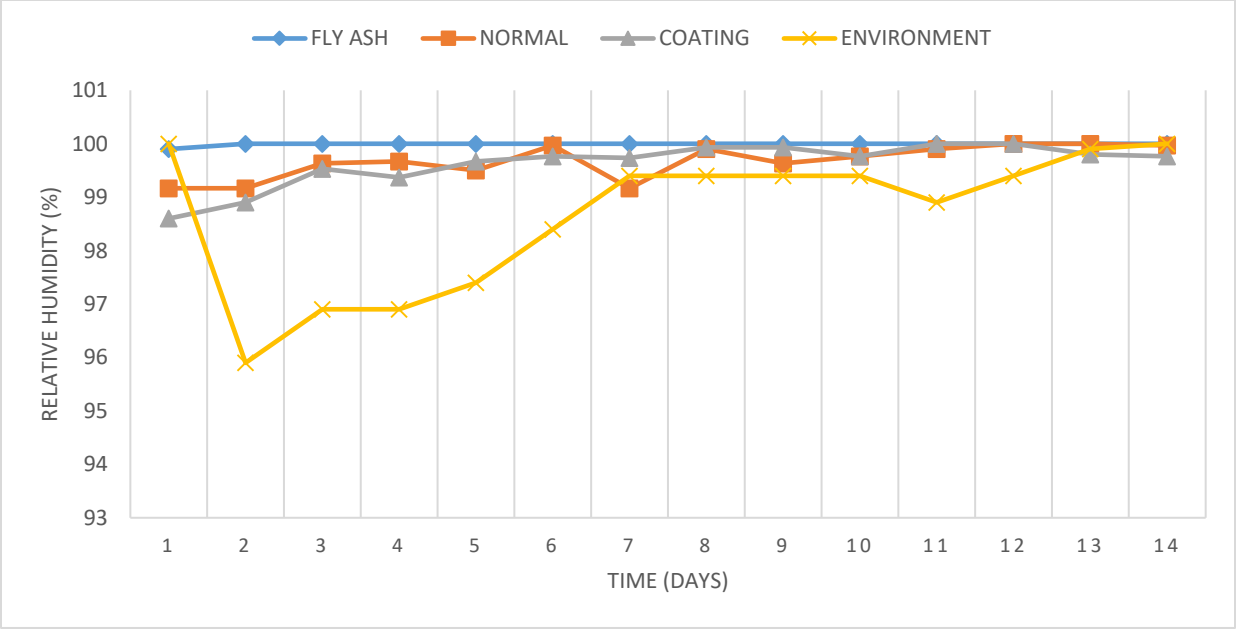


Figure 4.12 Internal relative humidity at Point 3 when samples were exposed to 100% relative humidity

It was expected that the relative humidity at points 2 and 3 should be the same as they were at the same depth and the ends of all the samples are coated and only loss of moisture would be possible through the surface only, this was observed in figure 4.13 the maximum difference between the relative humidity for point 2 and 3 is just 1% well with the accuracy of the sensor.

It can be observed clearly that the relative humidity for samples with fly ash have an approximately 100% relative humidity all through the 14 days and at the end of the 14 days, all the other samples with coating and without fly ash tend to have higher relative humidity compared to day 1 which shows that the coating is not that effective in preventing ingress of moisture in the early days after curing.

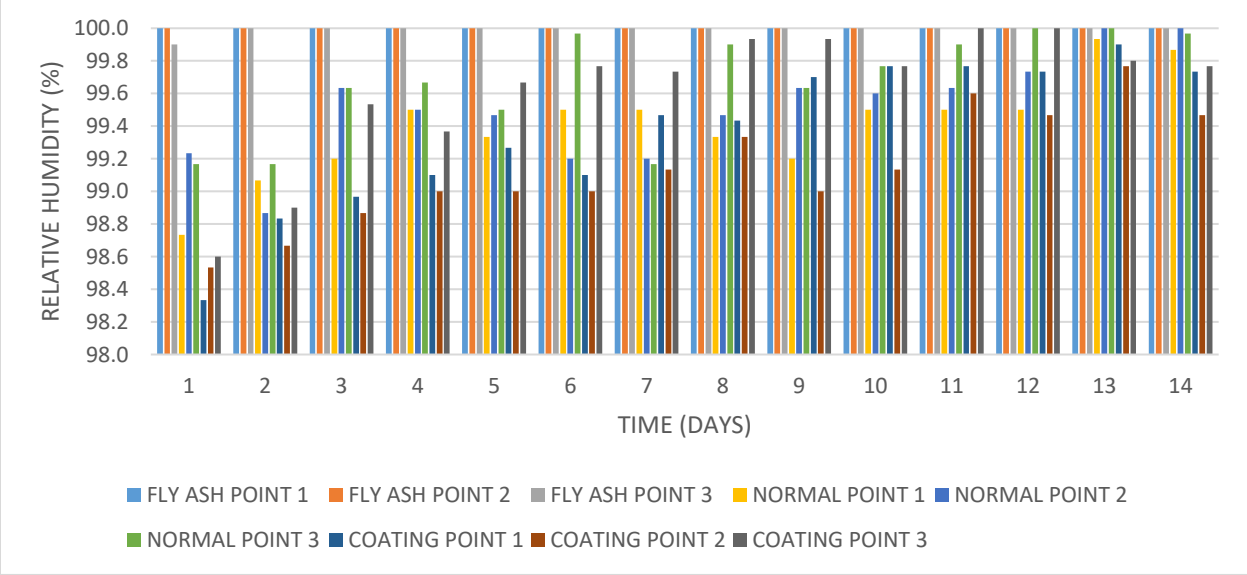


Figure 4.13 Internal relative humidity at all points when samples were exposed to 100% relative humidity

Since concrete is a complex structure and the overall moisture conditions cannot be determined based on the internal relative humidity measured at one point, the average of the internal relative humidity measured in each mixture and all points in the samples was calculated to have an idea of

the overall moisture condition of the whole sample as shown in figure 4.14. Again, it can be seen that the coated mixture maintained the lowest internal humidity while the fly ash mixture had the highest relative humidity.

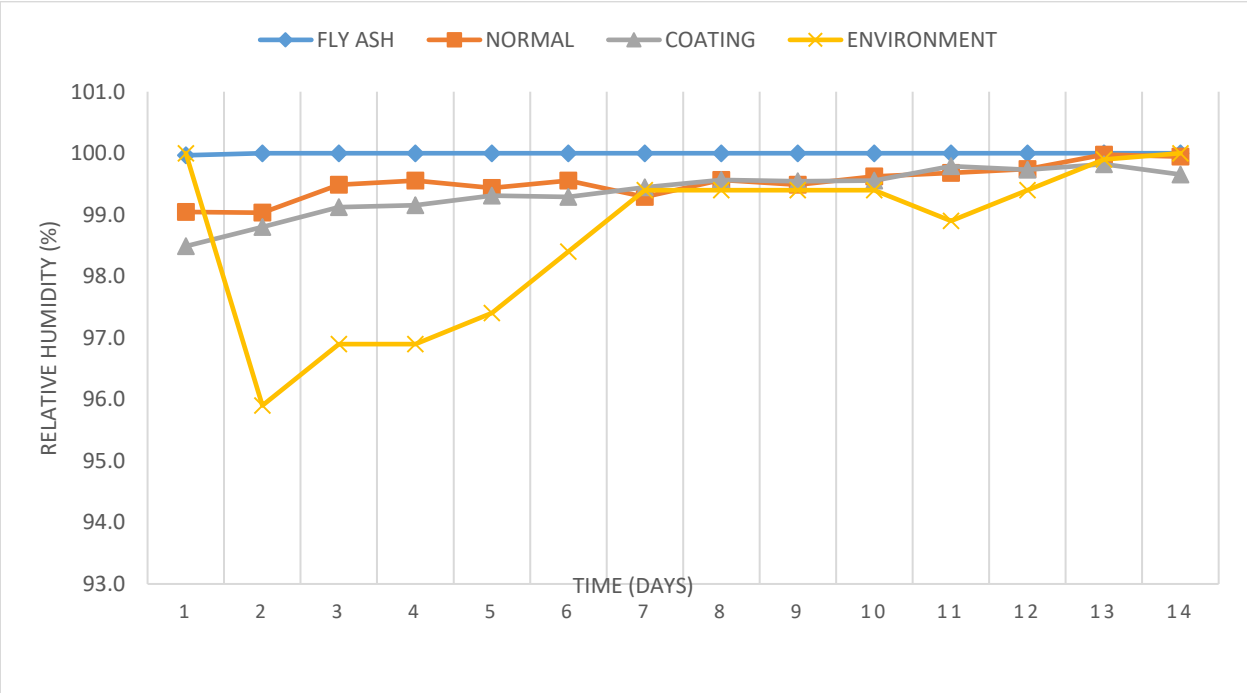


Figure 4.14 Average internal relative humidity when samples were exposed to 100% relative humidity

It was expected that the relative humidity for each group of samples will have approximately the same internal relative humidity at point 2 and 3 in the range of ± 2 % (as observed by Monfore (1963) for repeatability of relative humidity measurements in similar concrete sample) because both points are 25 mm away from the surface. This was observed in all the group of the samples as the internal relative humidity measured was approximately the same for point 2 and point 3 as shown in figures 4.11 and 4.12.

After 14 days of exposing the samples to 100% relative humidity, the samples were transferred into an environmental chamber with temperature configured at 23°C and relative humidity at 75% and the results for each point for each group of samples shown in figures 4.15 to 4.17. Due to lower efficiency of the environmental chamber, it was observed that the exact required 75% relative humidity was not constant for the duration of the test.

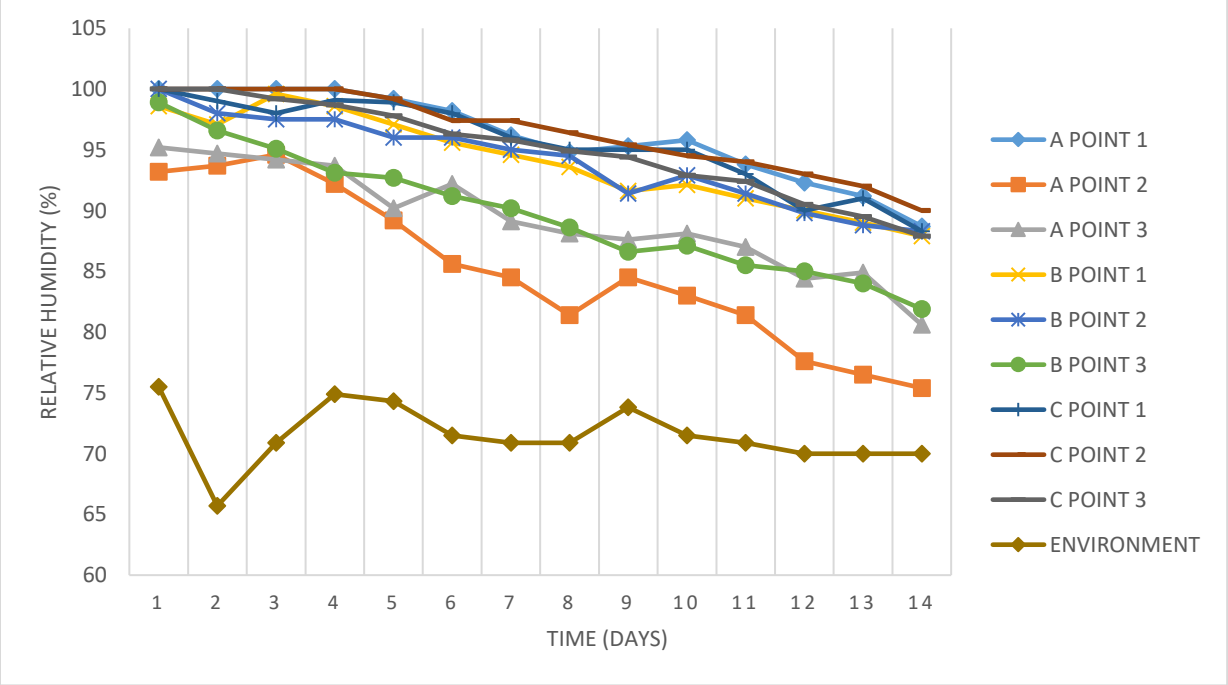


Figure 4.15 Internal relative humidity when exposed to 75% relative humidity in samples with 50% Fly ash

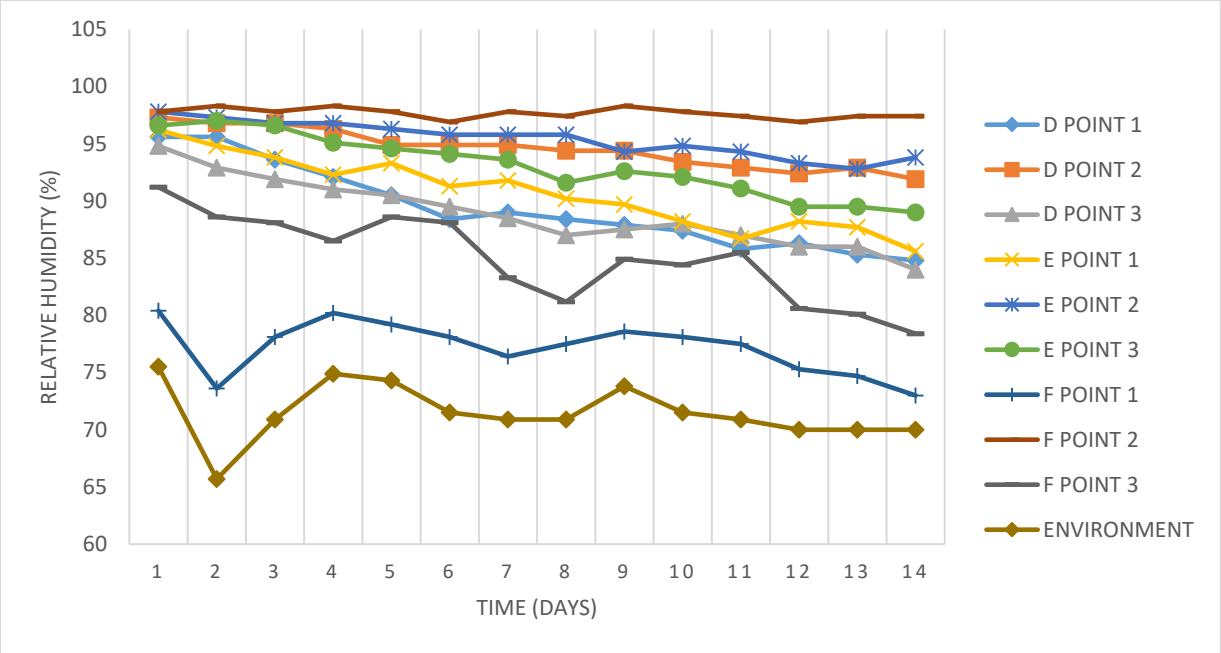


Figure 4.16 Internal relative humidity when exposed to 75% relative humidity in samples without SCM

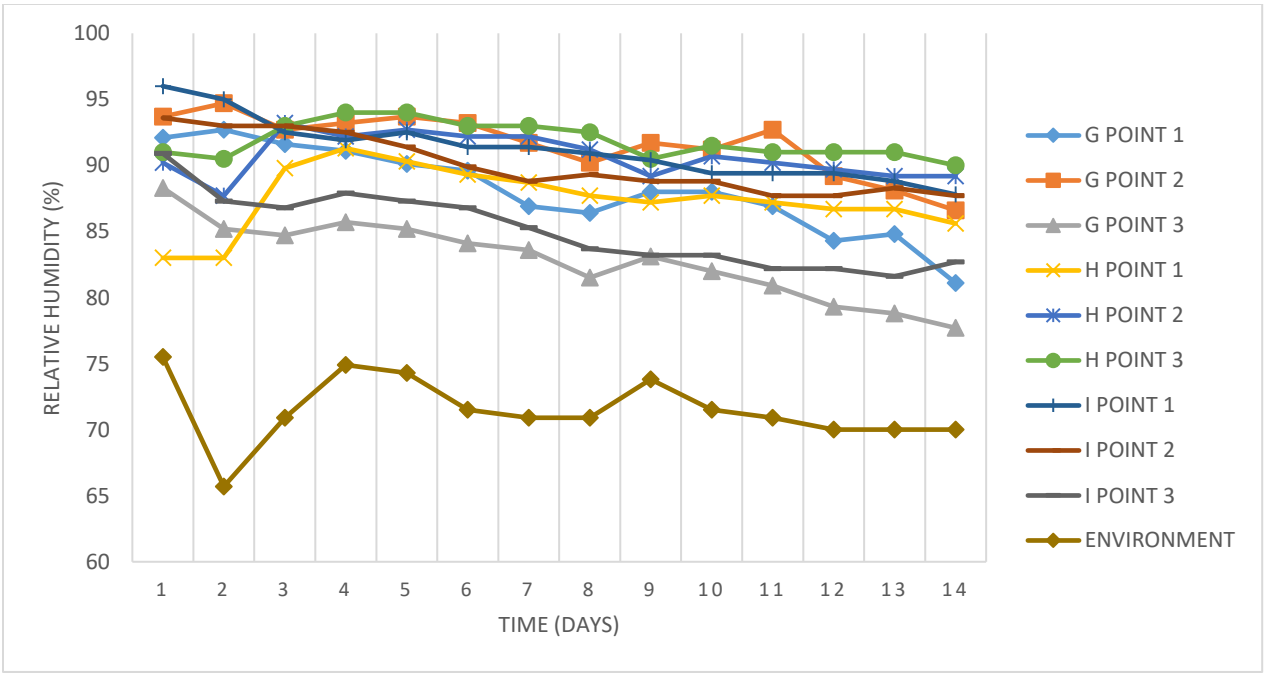


Figure 4.17 Internal relative humidity when exposed to 75% relative humidity in samples with coating

Figures 4.18 to 4.20 show the variation in the environmental relative humidity and the internal relative humidity measured at each of the 3 points in each of the samples. The environmental relative humidity was also measured each day and included on each figure. It can be seen that the internal relative humidity decreases over the 14 days, but does not come into equilibrium with the external conditions.

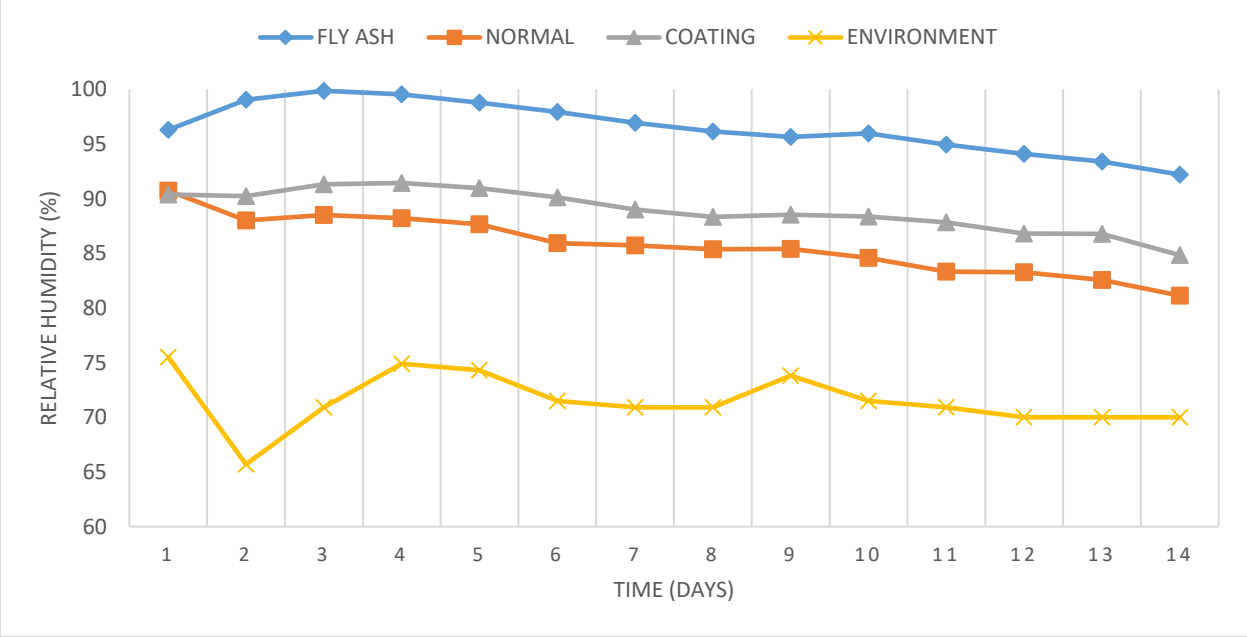


Figure 4.18 Internal relative humidity at Point 1 when samples were exposed to 75% relative humidity

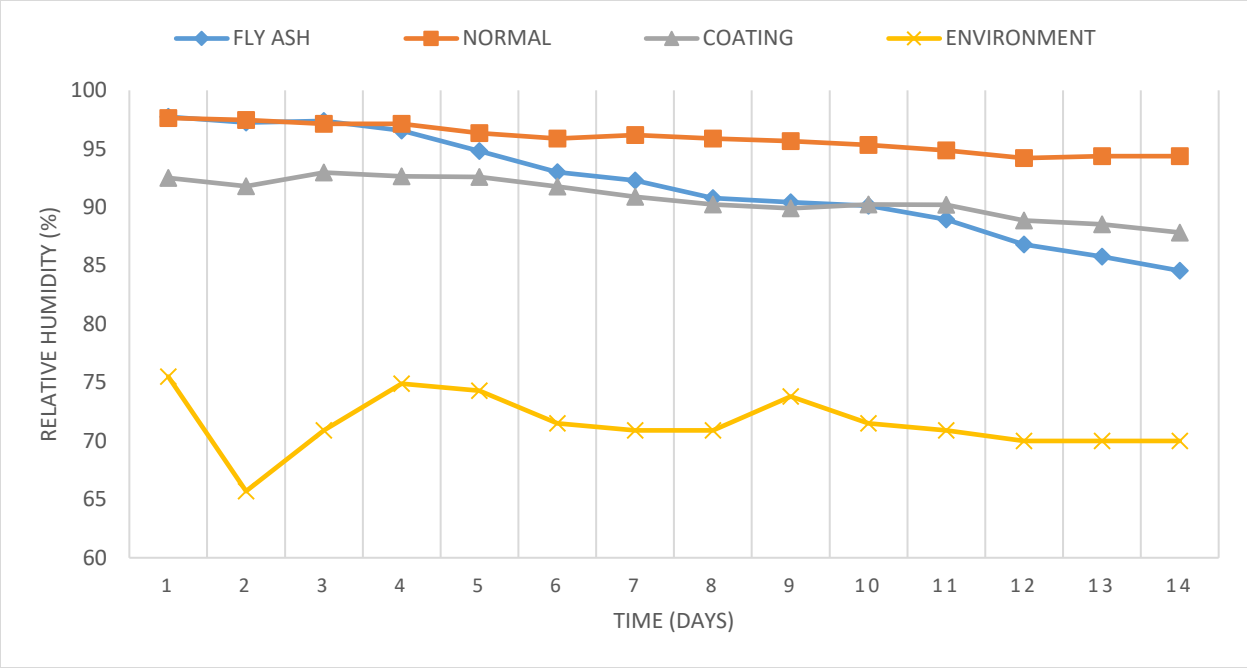


Figure 4.19 Internal relative humidity at Point 2 when samples were exposed to 75% relative humidity

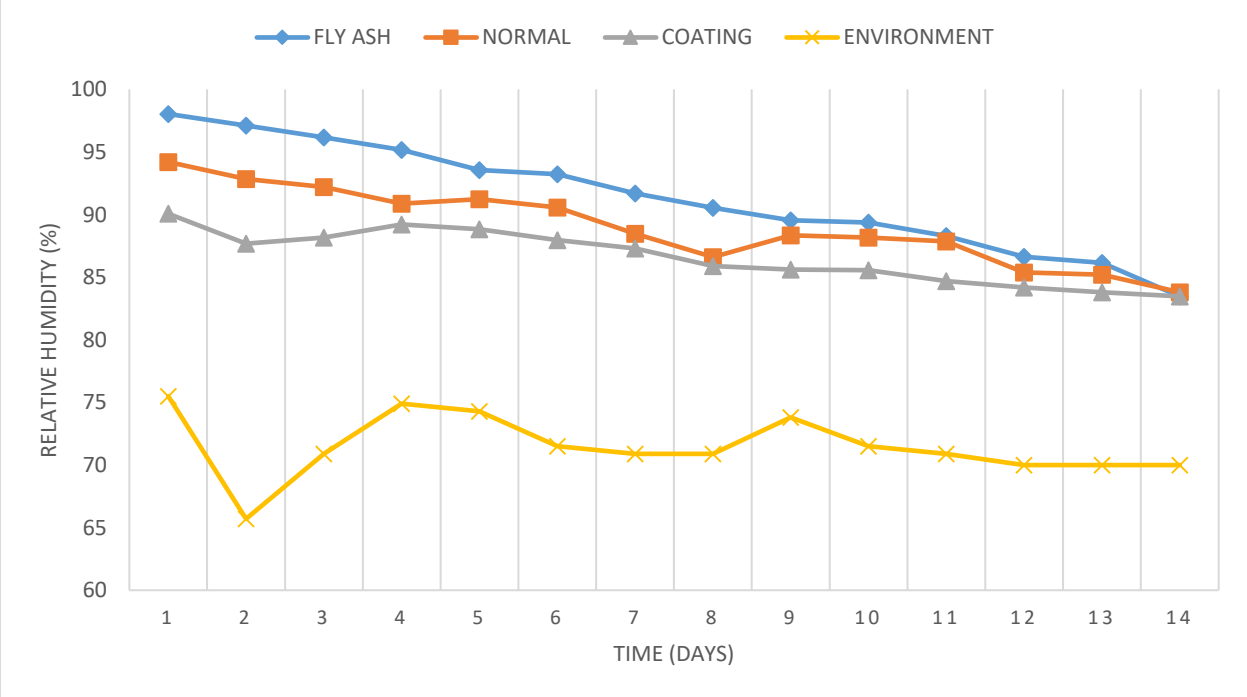


Figure 4.20 Internal relative humidity at Point 3 when samples were exposed to 75% relative humidity

It can be observed for each group of samples at point 1 (Figure 4.18) which is 25mm inwards that samples with fly ash have a higher relative humidity all through the duration of test compared to samples without fly ash and coating, but this was not the case at point 2 (Figure 4.19) which was 50 mm into the sample as the average relative humidity for samples with fly ash drops at day 4 and was the lowest at 14 days. At point 3 (Figure 4.20), it was observed that samples with fly ash have a higher relative humidity at the beginning of the test but reduce with time and have almost the same relative humidity at the end of the 14 days. Though it was expected that for each group of samples they will have approximately the same relative humidity at point 2 and 3, only samples with fly ash show this as the maximum difference between the point 2 (Figure 4.19) and point 3 (Figure 4.20) observed was 1%. In order to have a true representation of the relative humidity inside the samples, the average of the relative samples for each group of samples were determined and shown in figure 4.21, it was observed that samples with fly ash have a higher internal relative humidity compared to samples with coating and samples with neither coating or fly ash.

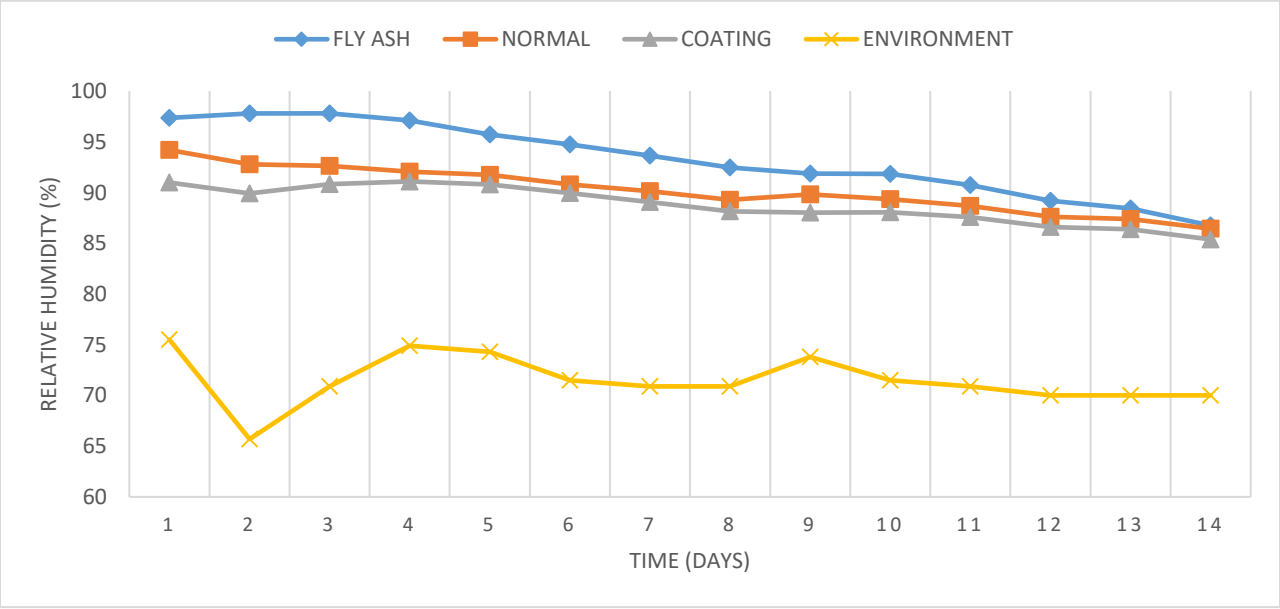


Figure 4.21 Average internal relative humidity when samples were exposed to 75% relative humidity

After 14 days of exposure the samples to 75% relative humidity, the environmental chamber was reconfigured to produce a relative humidity of 65% but the same fixed temperature as before so as to see the effect of lowering the relative humidity on the internal relative humidity inside the samples. The results are presented as follows and same measurement and comparisons made in the previous tests were made. Figures 4.22 to 4.24 show the measured internal relative humidity at each of the samples at all points and figures 4.25 to 4.27 shows the average internal relative humidity for each group of concrete samples with the same properties at each point for all the three points. To get the overall moisture condition of the samples with different properties in terms of the internal relative humidity, the average of internal relative humidity at all point in all samples with the same properties was calculated and presented in figure 4.28 i.e. for example; the average of all measured internal relative humidity at all point for all samples with fly ash was calculated to show a representation of the moisture content in the sample in terms of relative humidity, this was also done for samples with coating and samples without fly ash or coating.

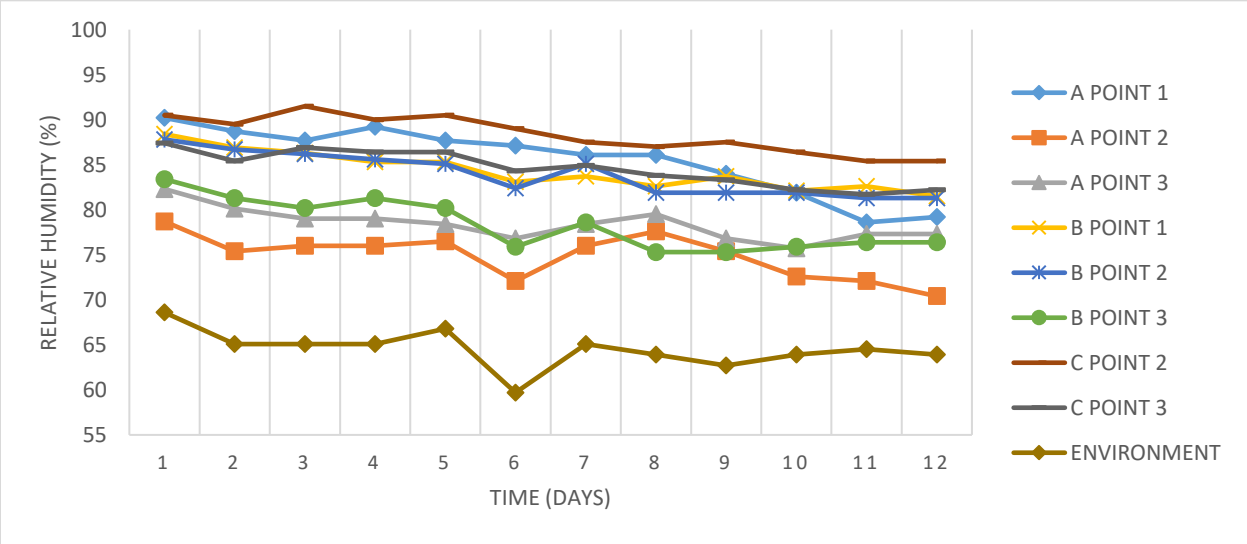


Figure 4.22 Internal relative humidity when exposed to 65% relative humidity in samples with 50% Fly ash

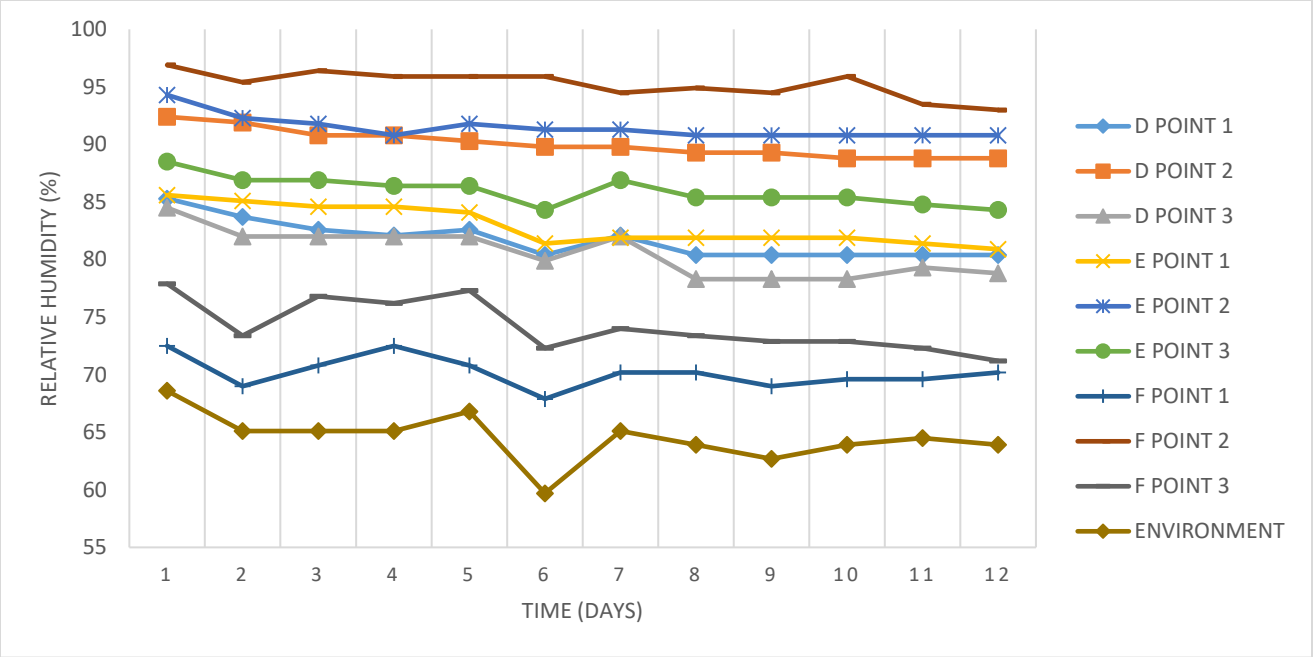


Figure 4.23 Internal relative humidity when exposed to 65% relative humidity in samples without SCM

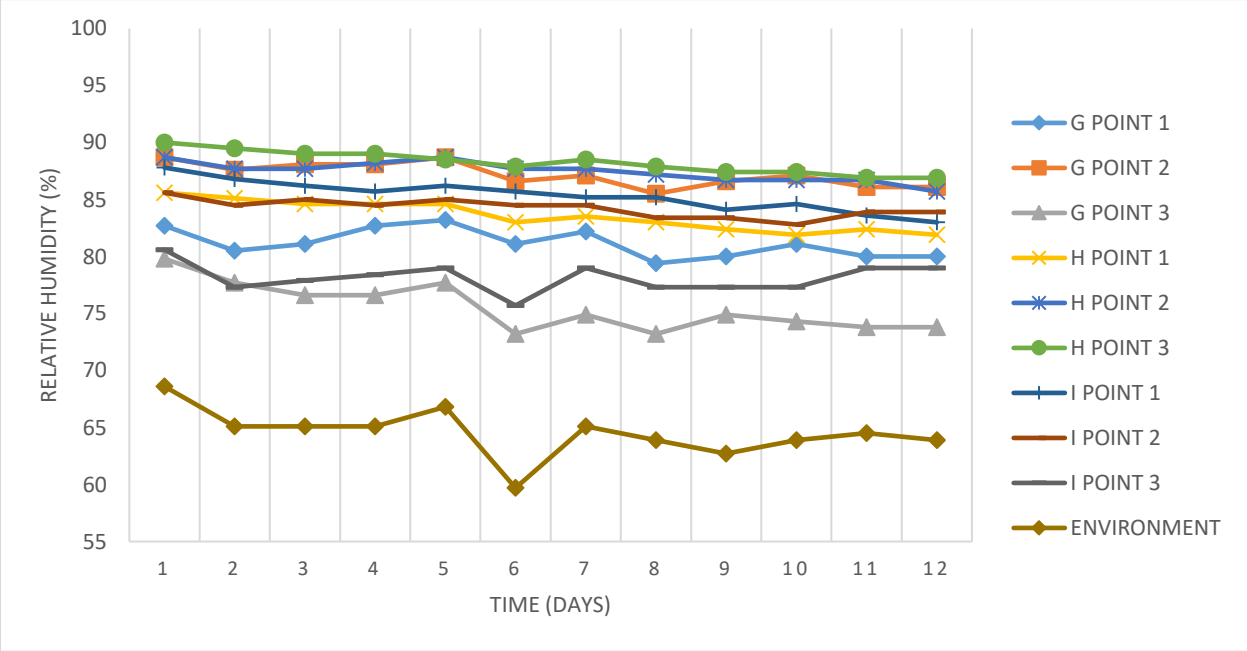


Figure 4.24 Internal relative humidity when exposed to 65% relative humidity in samples with coating

Figures 4.25, 4.26 and 4.27 show the average internal relative humidity measured for each group of samples at each point with figure 4.25 showing the measured internal relative humidity at point 1 for all samples in each group and figures 4.26 and 4.27 show for average internal relative humidity for points 2 and 3, respectively. There was a significant drop in the relative humidity of all samples on the 13^{th} day which might be as a result of the effectiveness of the salt used. Therefore, the conclusions from this part of experiment was based on the results of the 12^{th} day. This drop in relative humidity was not noticed in the environmental relative humidity which might be because the sensors record the result once daily and at the time the environment's humidity was taken, there has not been any drop yet. From the average of the internal relative humidity (Figure 4.28), it can be seen that samples with coating and samples with neither coating or fly ash have approximately the same internal relative humidity throughout this test. The fly ash samples start with a slightly average RH, but decrease at a more rapid rate ending with a lower RH than the other mixtures. Though there was a drop in relative humidity at all point in all the samples, the relative humidity at all the points were greater than the environment's relative humidity in the 12 days considered.

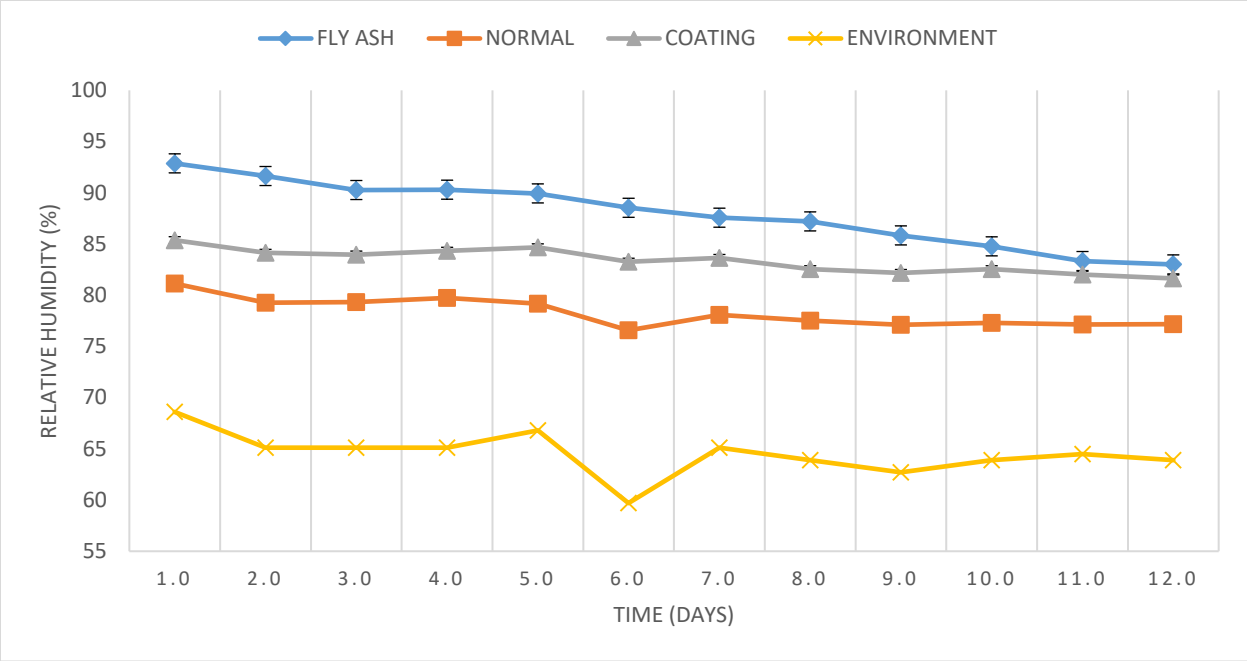


Figure 4.25 Internal relative humidity at Point 1 when samples were exposed to 65% relative humidity

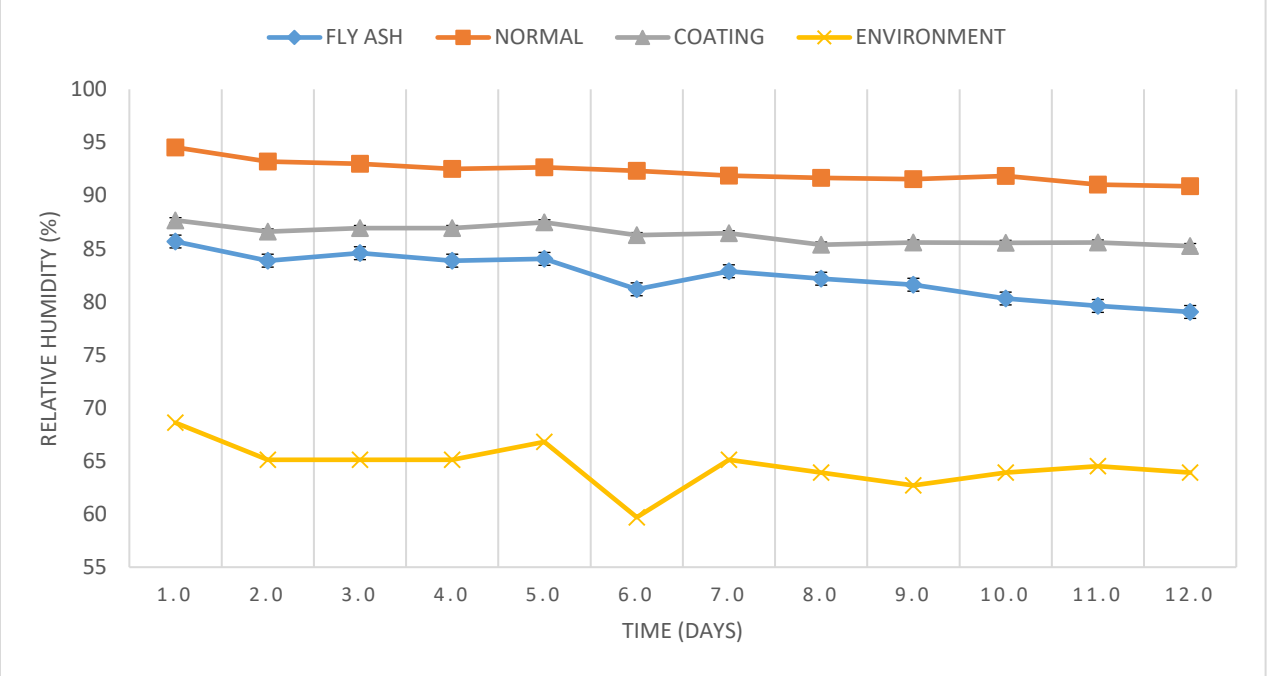


Figure 4.26 Internal relative humidity at Point 2 when samples were exposed to 65% relative humidity

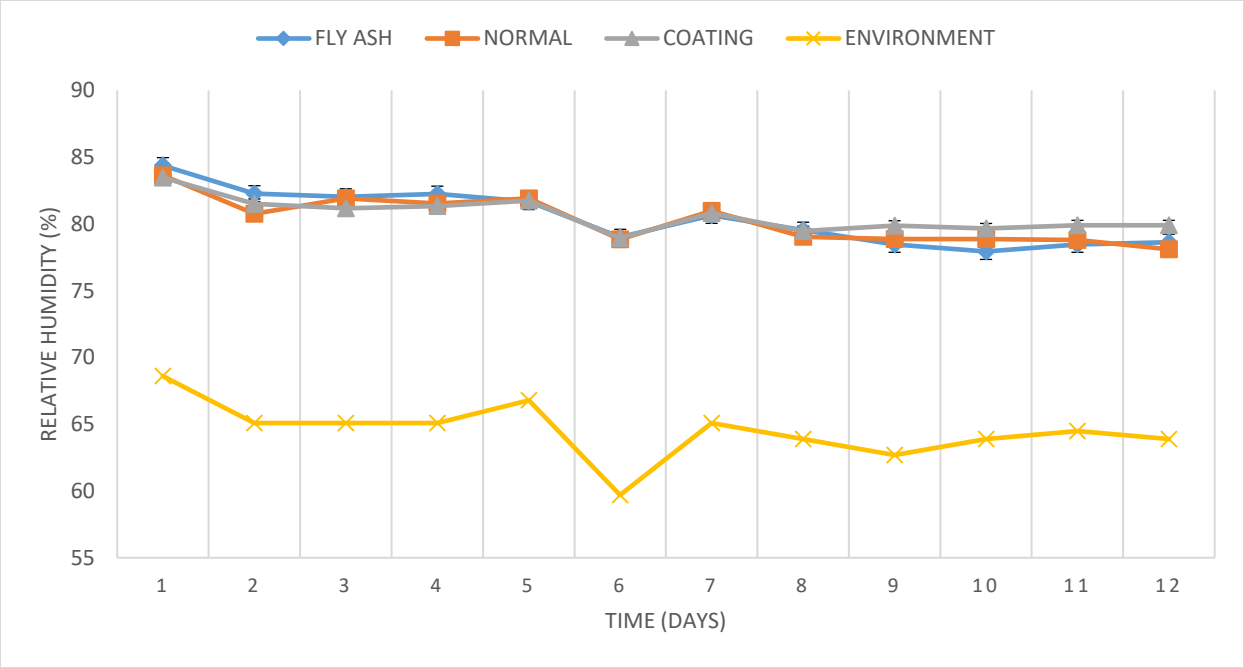


Figure 4.27 Internal relative humidity at Point 3 when samples were exposed to 65% relative humidity

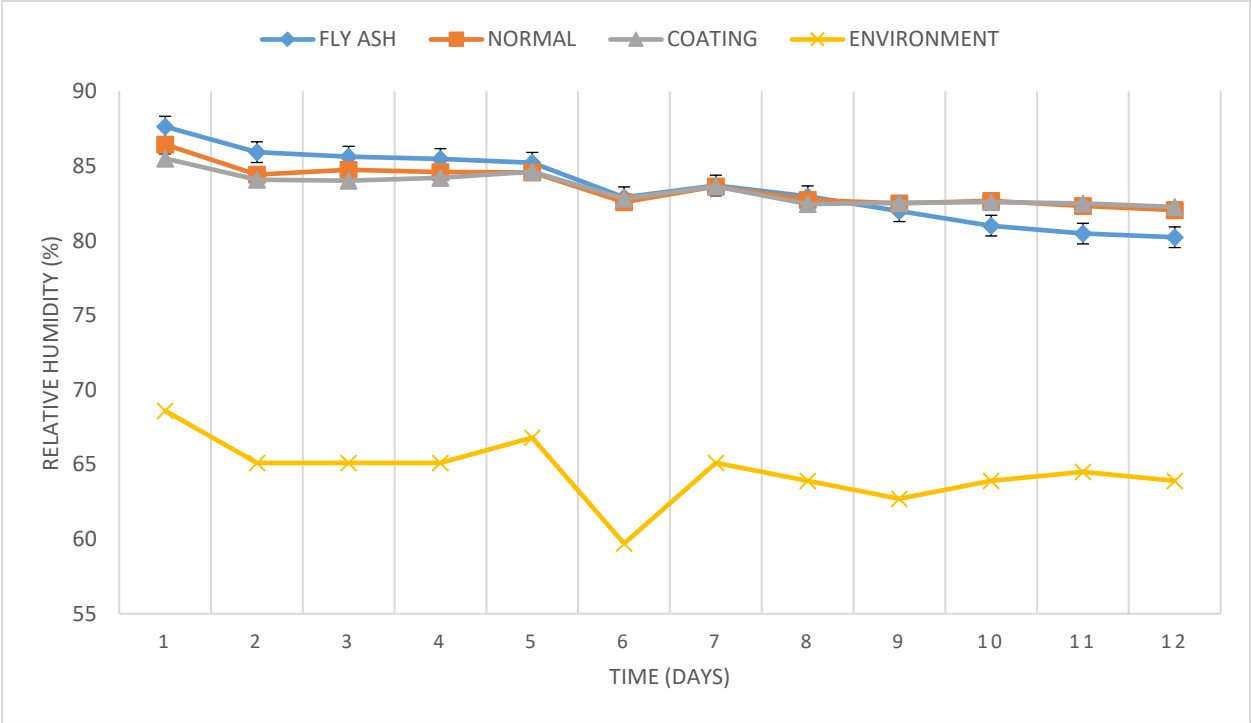


Figure 4.28 Average internal relative humidity when samples were exposed to 65% relative humidity

After exposure of the concrete samples to 65% for 14 days, they were further exposed to a higher relative humidity of 95% to see the effect of increase in the external relative humidity on internal relative humidity measured in the concrete samples. The environmental chamber was also used for this condition and it should be noted that all these measurements are done at a fixed temperature so as to understand the effect of the external relative humidity. The results shown in figures 4.29 to 4.35 are for samples exposed to an average relative humidity of 95% at room temperature. Some variations were observed in the external relative humidity as a result of the environmental chamber which was unable to give a constant relative humidity of the duration of the test. A sensor was placed in the chamber to measure the actual conditions to which the samples were exposed. Figures 4.29 to 4.31 shows the measured internal relative humidity at each point in the concrete samples for each group of samples with the same properties i.e. with 50% fly ash, coating and without coating or fly ash, figures 4.32 to 4.34 shows the measured internal relative humidity obtained for all the samples at each specific point in the sample. Figure 4.35 shows the average relative humidity at all points in each group of concrete samples with the same properties.

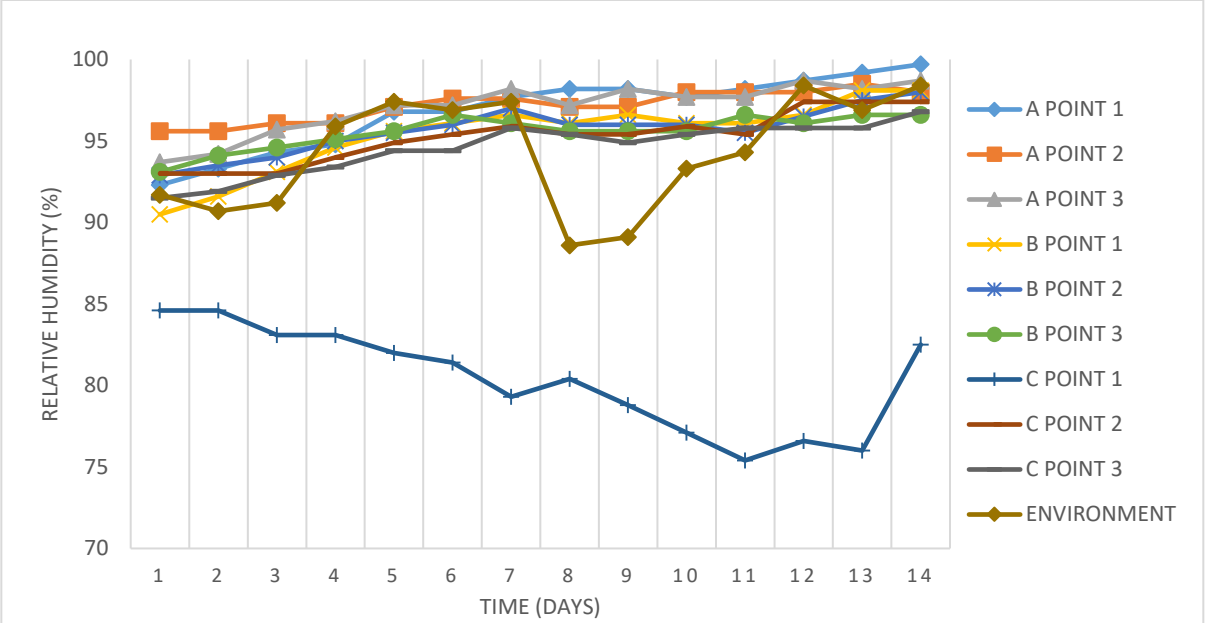


Figure 4.29 Internal relative humidity when exposed to 95% relative humidity in samples with 50% Fly Ash

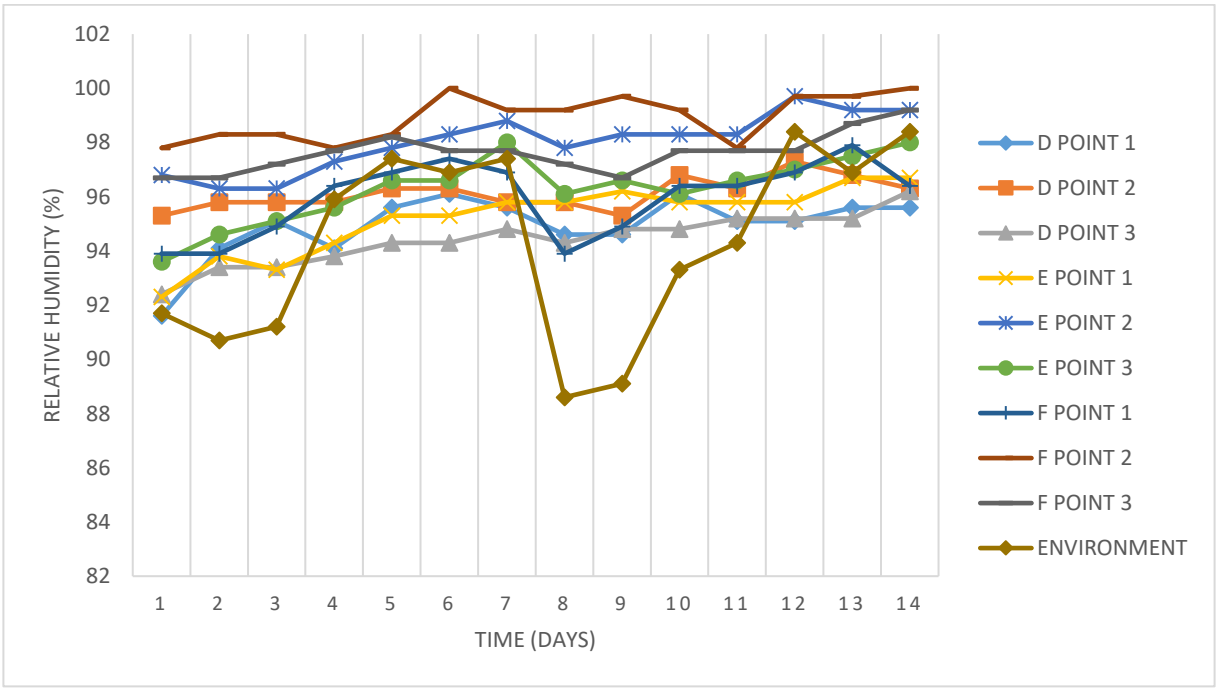


Figure 4.30 Internal relative humidity when exposed to 95% relative humidity in samples without SCM

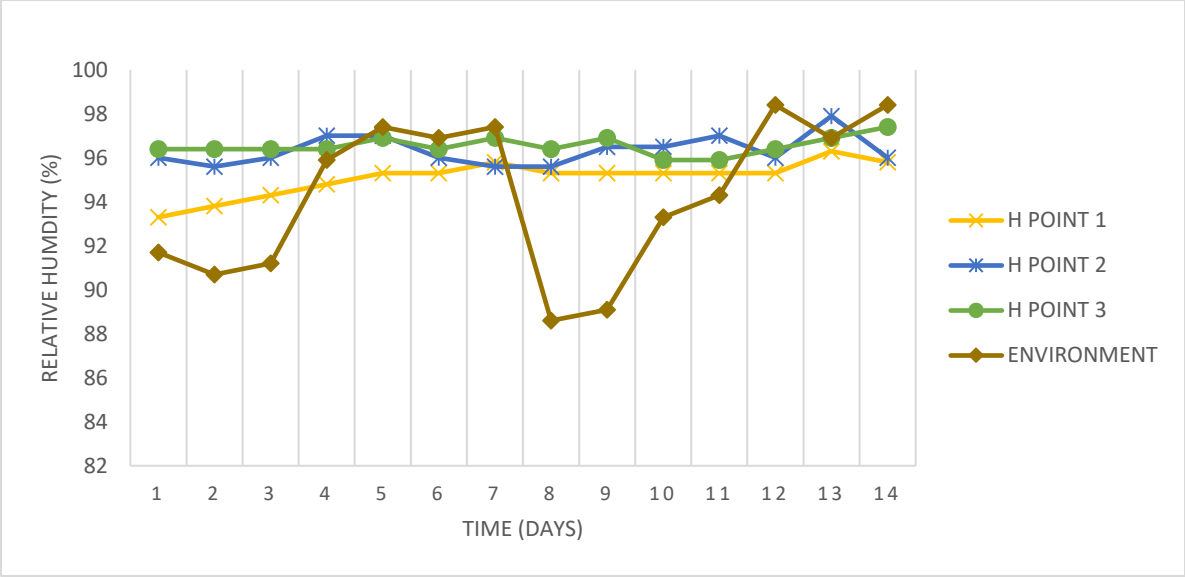


Figure 4.31 Internal relative humidity when exposed to 95% relative humidity in samples with coating

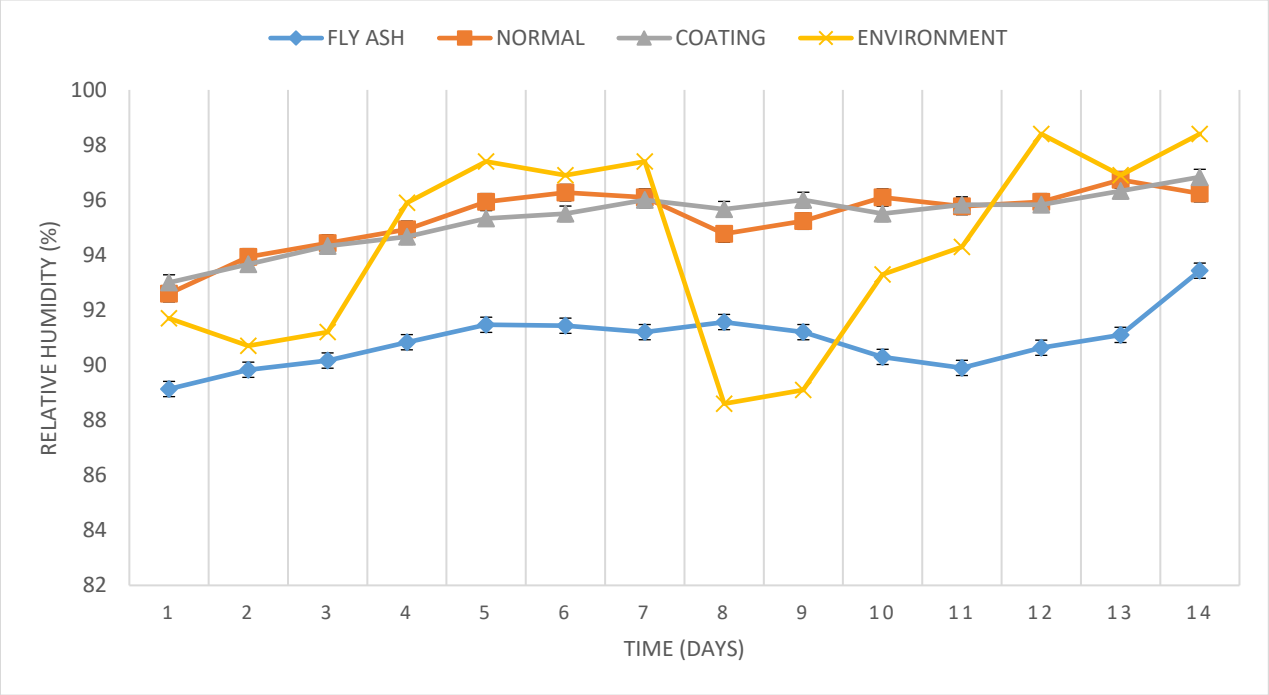


Figure 4.32 Internal relative humidity at Point 1 when samples were exposed to 95% relative humidity

It was observed that the relative humidity at all points increased within 24 hours of putting the samples in a higher relative humidity environment, this might be as a result of the small size of the sample, this might not be the case with large structures or a possibility of leakage at the stoppers which might have resulted in the increased relative humidity measured. From all the samples; point 1 has a higher increase in relative humidity compared to other points for each group of samples. Point 1 of samples with 50% fly ash had approximately 6% increase in relative humidity, point 2 and 3 with approximately 4% increase in humidity. For samples with coating; point 1 had approximately 11% increase in relative humidity while point 2 having an increase of approximately 10%. and point 3 having an approximate increase of 6% in relative humidity. For samples without coating or fly ash, point 1 had an increase of approximately 20%, and point 2 and 3 with an approximate increase of 6% in relative humidity.

It can be observed from figures 4.29 to 4.31 that the recorded average relative humidity measured at point 1 for all samples was lower compared to point 2 and 3 for the first several days. This is an expected result as point 1 which is 25mm depth from the surface which is closer than points 2 and 3 which are 50mm from the surface of the sample. The humidity was lowered in the previous environment. It can also be observed from figure 4.32 that samples with fly ash have a lower relative humidity compared to samples with coating and samples with neither coating nor fly ash. At the end of the test period, the average relative humidity in samples with 50% fly ash was lower compared to other samples with samples with neither coating nor fly ash having a higher increase in relative humidity compared to other group of samples. Also from figure 4.35, it was observed that samples with coating and samples with neither fly ash nor coating have approximately the same relative humidity higher than the samples with fly ash.

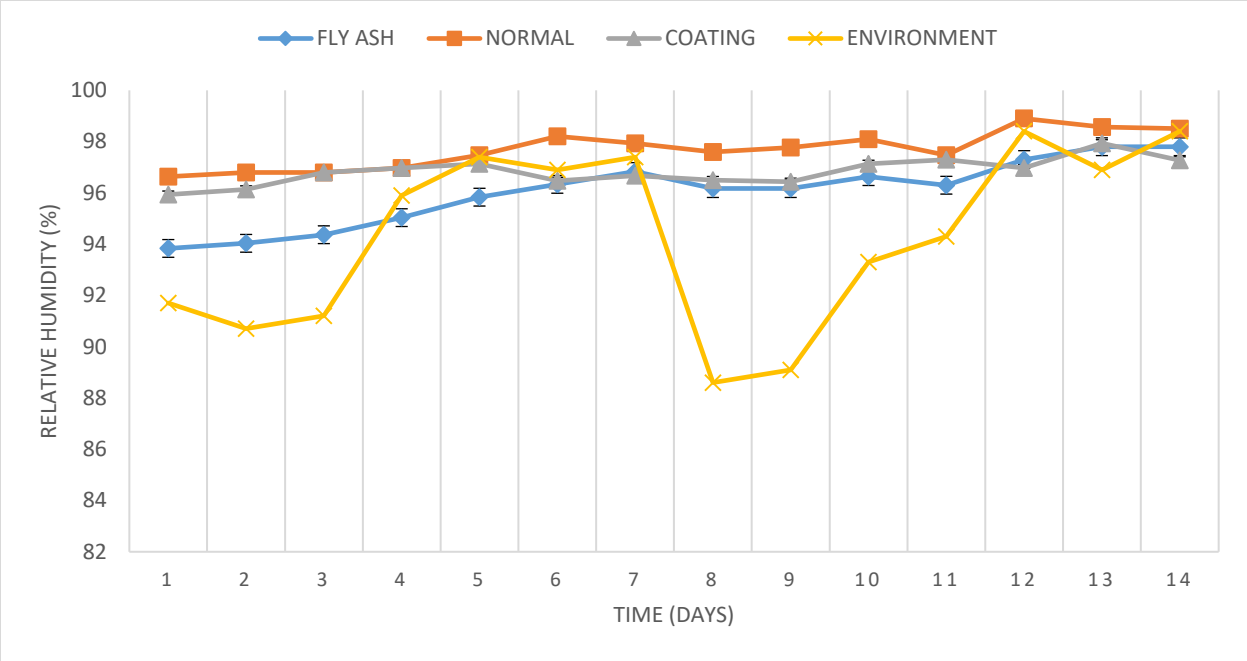


Figure 4.33 Internal relative humidity at Point 2 when samples were exposed to 95% relative humidity

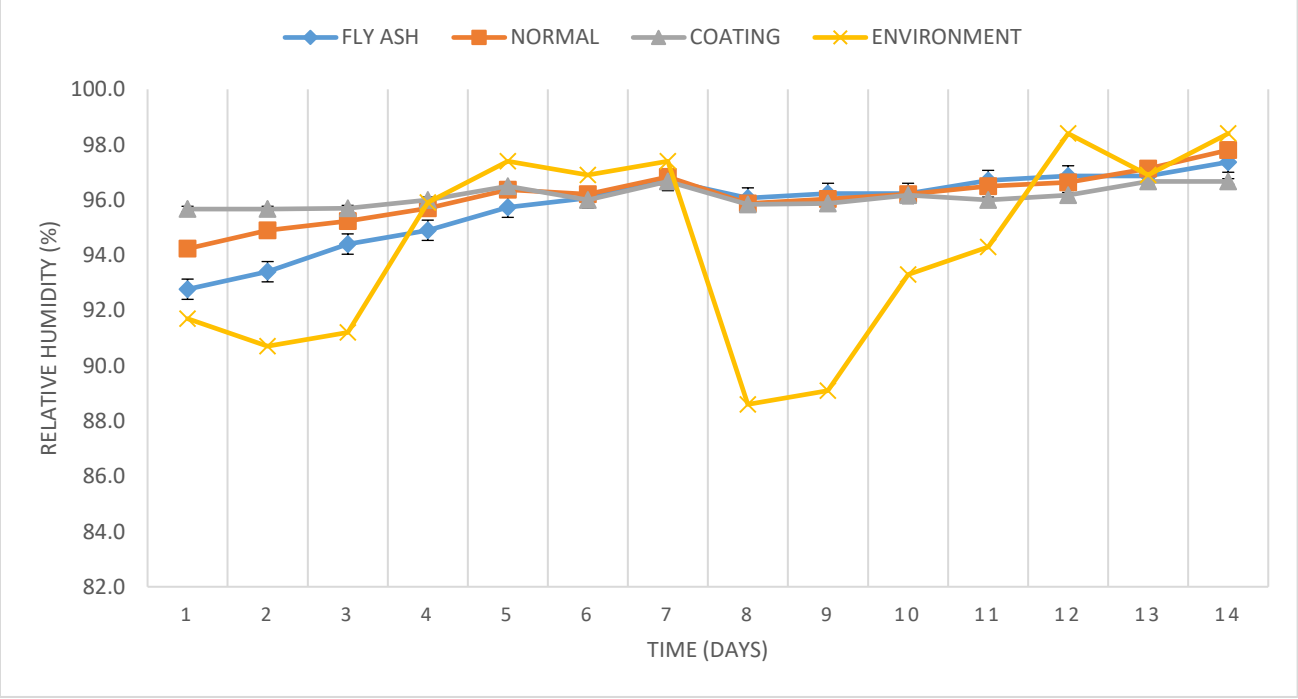


Figure 4.34 Internal relative humidity at Point 3 when samples were exposed to 95% relative humidity

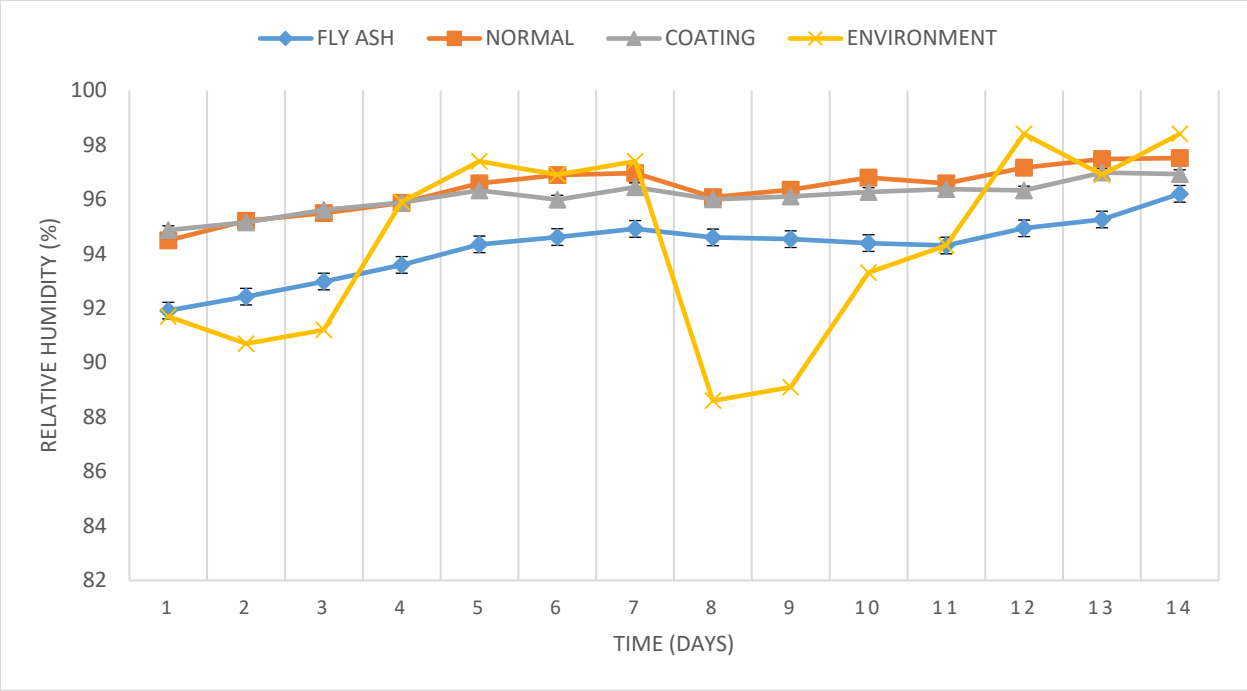


Figure 4.35 Average internal relative humidity when samples were exposed to 95% relative humidity

To have a broad view of the results at the different external relative humidities to which the samples were exposed, figures 4.36 to 4.38 show the combined average internal relative humidity measured for each group of samples.

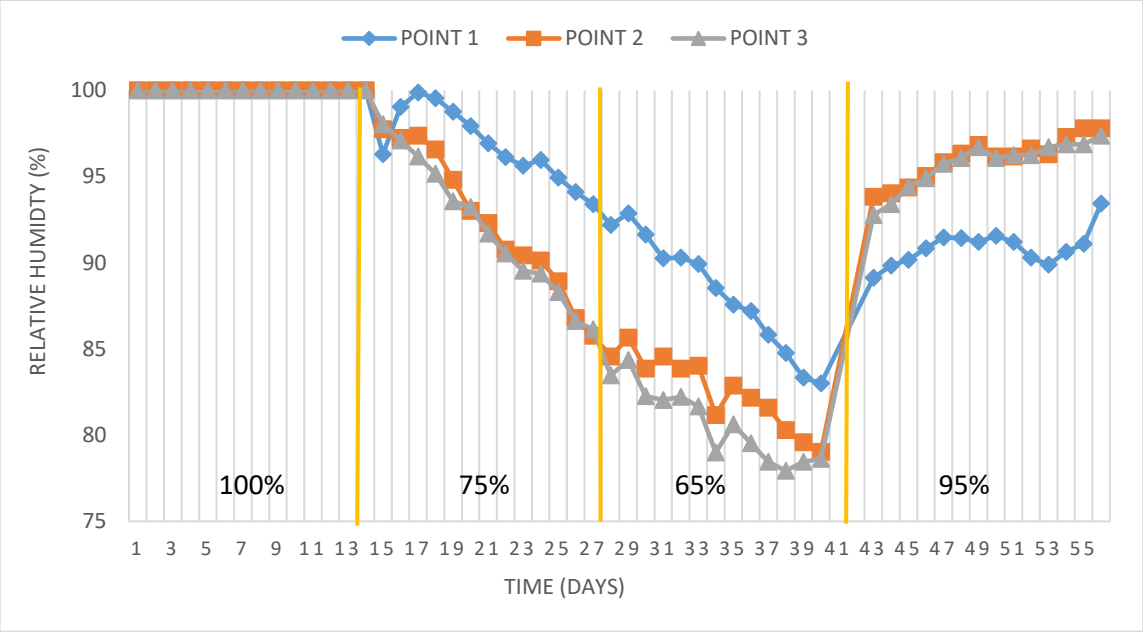


Figure 4.36 Average internal relative humidity at each point in samples with 50% fly ash

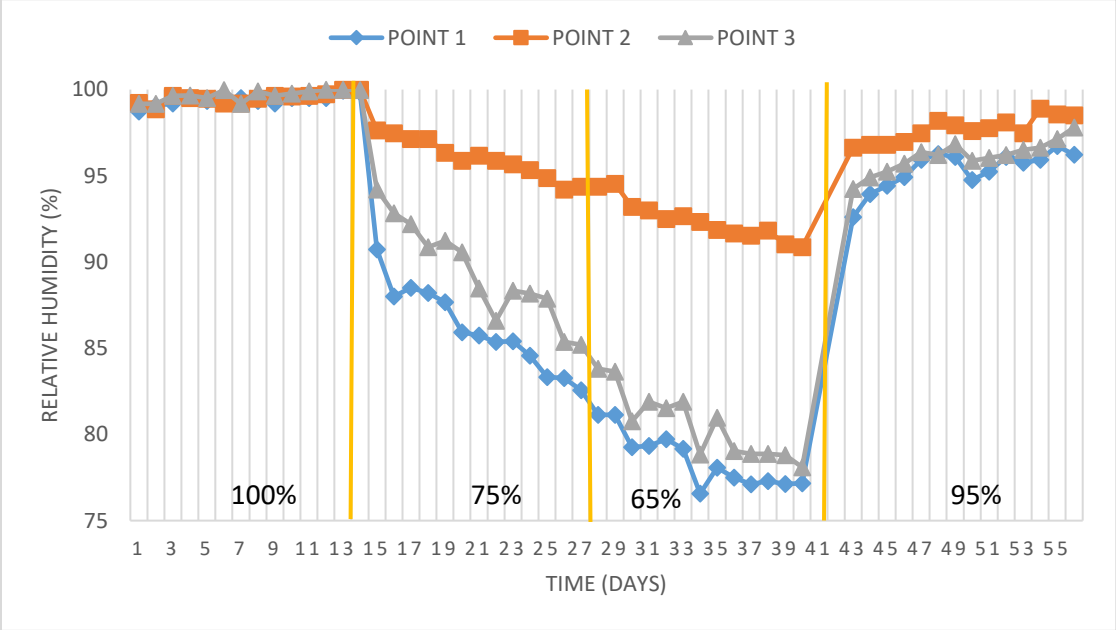


Figure 4.37 Average internal relative humidity at each point in samples with neither fly ash nor coating

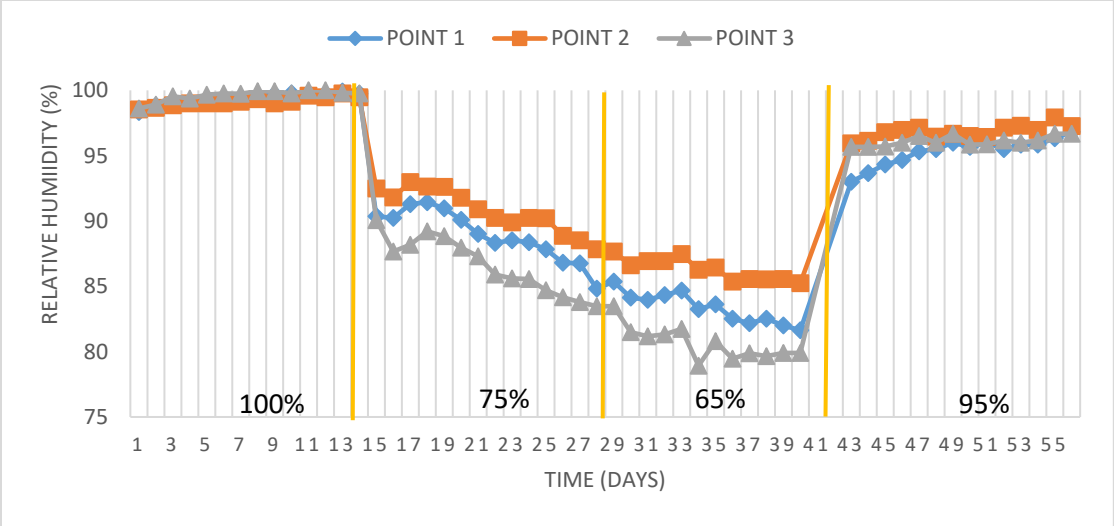


Figure 4.38 Average internal relative humidity at each point in samples with coating

From figures 4.36 to 4.38 which shows the average measured relative humidity in each group of samples; it would be observed that the measured internal relative humidity changes with change in external relative humidity with the measured internal relative humidity decreasing when placed in a lower relative humidity and increasing when placed in a higher relative humidity though the amount of decrease and increase varies with the composition of the concrete.

For samples with 50% fly ash (Figure 4.36), it would be observed that after placing the samples in two lower relative humidity (75% and 65%) and then placed in a higher relative humidity of 95%, the measured internal relative humidity at point 1 which is closer to the surface did not have a high decrease compared to normal samples and samples without coating, this will be as a result of the hydration of the fly ash making the pore system more compact and limiting the amount of ingress of moisture. It would still be observed from figure 4.36 that points 2 and 3 have a higher relative humidity compared to point 1 and this would be as a result of the moisture already in the sample not being able to go out easily too as a result of the hydration of fly ash.

It would be observed from figure 4.37 that samples with neither fly ash or coating, point 2 shows the highest humidity at all stages. The reason for the large difference is not known. The differences in humidity at points 1 and 3 showed large variations (up to 10-15%) between the three samples. Point 2 showed much less variation (up to 7%). See the Appendix for individual data points.

In the samples with coating, as shown in Figure 4.38, point 1 has intermediate average humidity compared to points 2 and 3. The measured internal relative humidity for samples with coating, it ca be observed that there's no significant effect of depth on the measured relative humidity at higher relative humidity of 100% and 95%. Differences appear at 75% and 65%, but these differences between the points are much less than the other mixtures. The coating was intended to provide a barrier to moisture transfer. It could be possible that the coating was not applied thick enough to provide a complete barrier.

From the results obtained by exposing the samples to various relative humidity, the following can be observed:

- There's no significant effect of depth on the internal relative humidity measured in samples with coating especially at higher relative humidity as the measured internal relative humidity measured in all samples with coating is in the range of ±2.5% at higher relative humidity

- Samples containing 50% fly ash maintains higher moisture at days after casting at the surface but as the hydration of the fly ash proceeds with time and the microstructure becomes denser, the samples with fly ash lowers the ingress of moisture into the concrete.

- Possibility of obtaining similar relative humidity in the range of ±2% at the same point in similar sample is more attainable at higher relative humidity than lower relative humidity as concrete has a very complex microstructure and the moisture of moisture at the same point in similar samples will vary.

4.2 PHASE 2

4.2.1 EXPERIMENT 2A

The following results are obtained to observe the effect of external relative humidity on the progression of alkali silica reaction. Figure 4.39 shows the expansion in terms of the percentage change in length of each sample for a period of 28 days after removal from sodium hydroxide and exposure to various humidities. Samples A, B and C are the samples exposed to 100% relative humidity, samples D, E and F are the samples exposed to 75% relative humidity, samples G, H and I are the samples exposed to 60%. As stated in chapter 3, surface area of the samples was observed after initial soaking for 14 days in sodium hydroxide solution and the average of surface area expansion for each group of relative humidity exposure is shown in figure 4.40. To see the correlation in mass gain of the samples as an indication of progression of ASR, the mass of the mortar samples was also taken alongside the surface area expansions to see the correlation and this is presented in figure 4.41 which shows the variation in masses for the period of time for all the classes of exposure conditions.

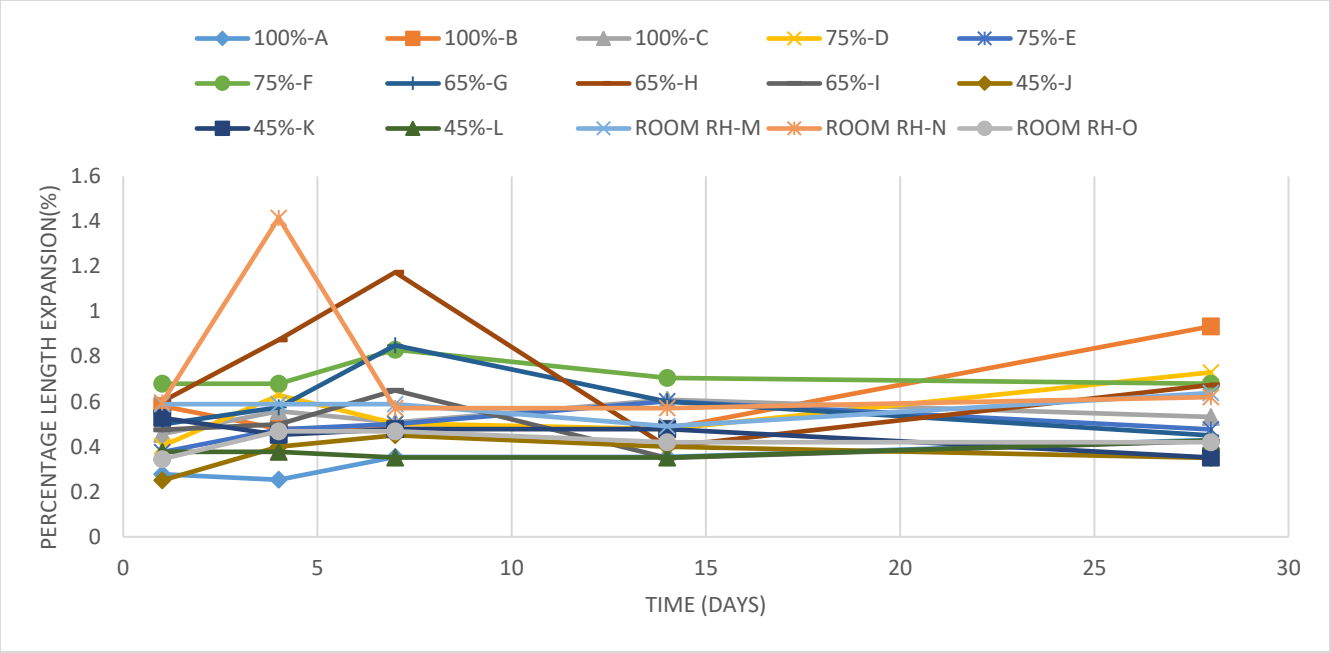


Figure 4.39 Length expansions of each mortar sample exposed to varying relative humidity at 38˚C after removal from NaOH solution

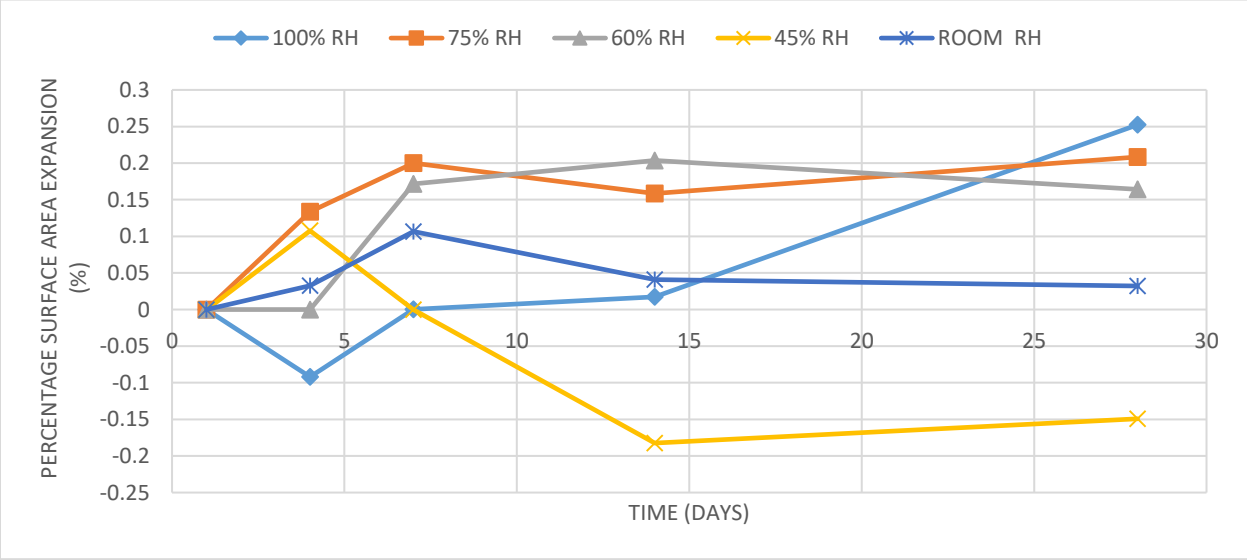


Figure 4.40 Surface Area Expansion of samples days after removal from NaOH solution to exposed to varying relative humidity

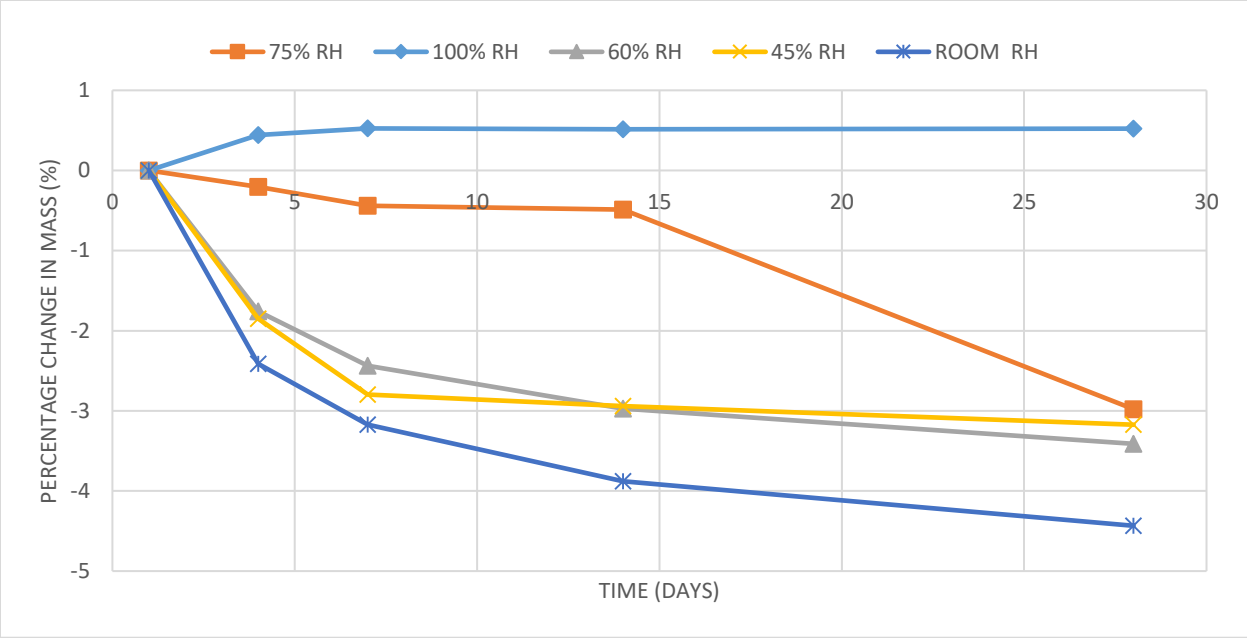


Figure 4.41 Percentage change in mass of samples days after removal from NaOH solution to exposed to varying relative humidity

It can be observed from figure 4.40 that within the first few days, the samples exposed to 100% relative humidity shrunk but the mass increased as it can be seen in figure 4.41, the surface area then increased afterwards having the highest expansion at the end of 28 days. It was also observed that it was only samples exposed to 100% relative humidity that have higher mass compared to one they were first exposed. Though the samples exposed to 45% relative humidity had higher surface area expansion in the first few days, the surface areas later reduced as time went by and these set of samples have lower surface areas compared to when they were first exposed.

From the results presented in the figure 4.40, it was observed that to an extent the external relative humidity has an effect on the expansion of samples due to ASR. Though these results cannot be conclusive yet until further studies are done comparing more relative humidity levels and having

a control sample (i.e. compare the surface area expansion of non-reactive aggregate exposed to the same external conditions). At the end of the observation period of 28 days after removal from the sodium hydroxide solution, the samples exposed to 100% relative humidity have the highest surface area expansion.

Based on the theory that as alkali silica reaction proceeds, there is an expected increase in mass. This theory was proven as shown in the figure 4.41; the masses of the samples were measured at the same time when the expansion of the samples were measured. It was observed that the samples exposed to 100% relative humidity and have higher surface area expansion as compared to others also had a gradual gain in mass. Also, the other samples in other exposure conditions showed a decrease in their mass which shows there is no continuation in the ASR due to lack of moisture. Though some expansions were observed for other external relative humidity apart from 100%, this might be as a result of the initial water present in the samples, and expansion stops when this is exhausted or lower than required to supply moisture to the alkali silica gel.

According to the literature, 80% relative humidity is the threshold commonly accepted for alkali silica reaction to occur. However, Ludwig (1989) suggested that the threshold for relative humidity should be assigned to a specific temperature, and Olafsson also showed in his studies a threshold of 80% at 23℃ and a threshold of 75% at 38℃, this was also observed in the results as shown on figure 4.40 that at 38℃, samples exposed to 75% and above expanded. Expansion was also observed in samples exposed to 60% relative humidity which might be as a result of small size of sample used. It was surprising that even samples exposed to a typical room relative humidity also expanded.

4.2.2 EXPERIMENT 2B

Following the procedure for ASTM C 1260, the following results are obtained using the cube mortar samples to determine the effect of water to cement ratio on ASR. Figure 4.42 shows the percentage length expansion for the average length measured for each mortar cube sample for 14 days. The average width expansion was also measured and it was used to calculate the surface area for each sample, the average percentage surface area expansion for each group of water to cement ratio is shown in figure 4.43. One of the cube samples for 0.55 w/c shows an exceptionally large expansion compared to all other samples which might be as a result of early expansion of a larger aggregate in the sample and the results for this sample was disregarded.

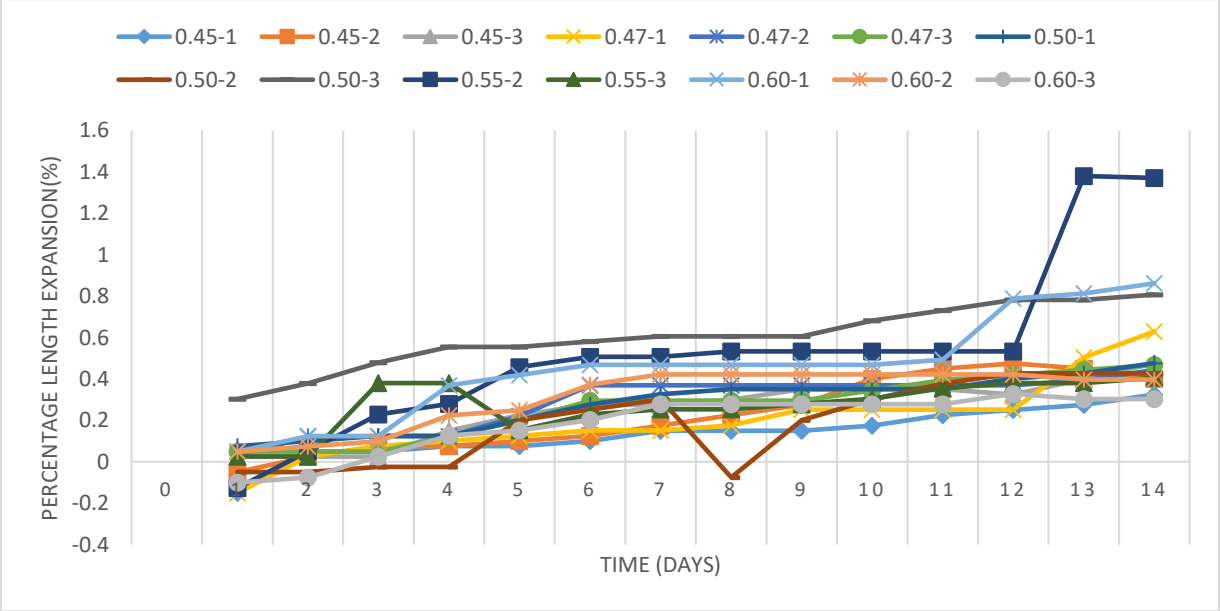


Figure 4.42 Percentage length expansion of all samples for 14 days

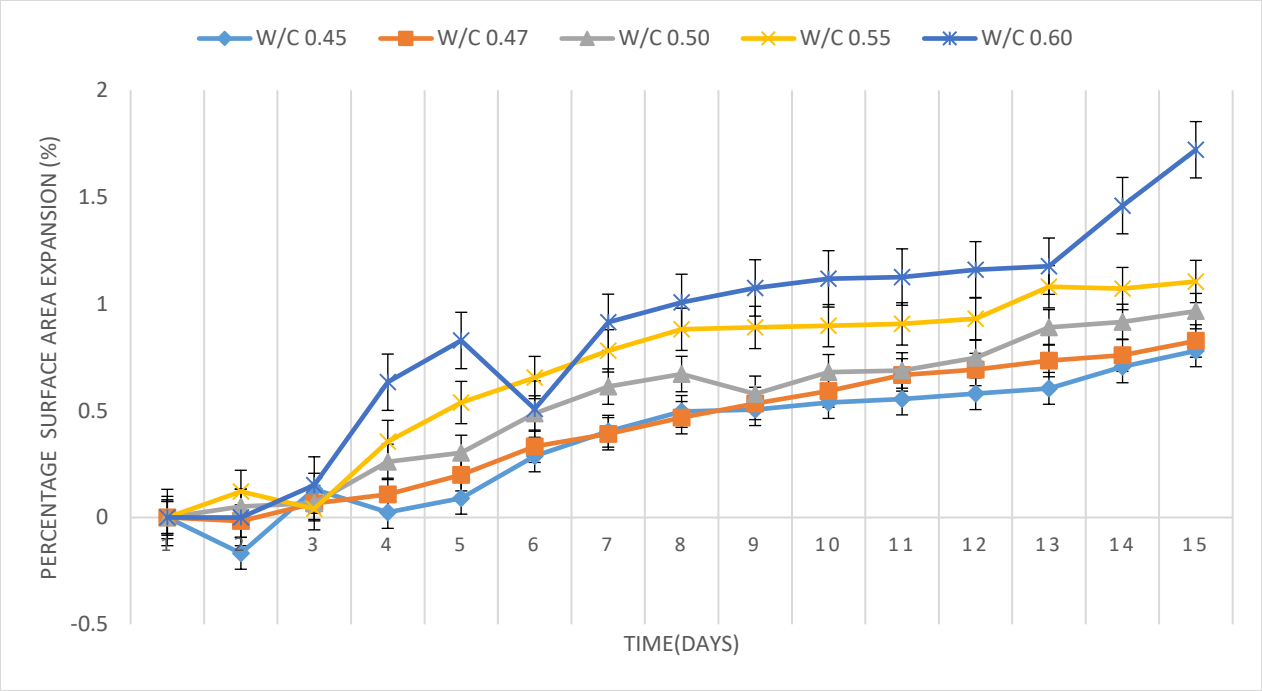


Figure 4.43 Percentage surface area expansion at 14 days

Figure 4.43 shows the 14 day average surface area expansion observed on the cube samples soaked in sodium hydroxide solution, it was observed that though the average surface area of the samples with 0.60 was lower on the 6th day which might be as a result of the gel being able to leak out of the sample thereby causing a temporary shrinkage, at the end of the 14 days it has the highest surface expansion area. It was also observed that the surface area expansion increases with the increase of water to cement ratio with from 0.45 up to 0.50 having similar expansions based on the standard error calculation made for the average of the surface area expansions.

After the 14 days of submerging the samples in sodium hydroxide; the samples were taken out and dried, then viewed under an optical microscope to observe the cracks caused as a result of the alkali silica reaction as these cracks are not visible to the human eyes. Figures 4.44 to 4.48 shows

the captured microscope images for all the five water to cement ratios that was tested, i.e. water cement ratios 0.45, 0.47, 0.50, 0.55 and 0.60.

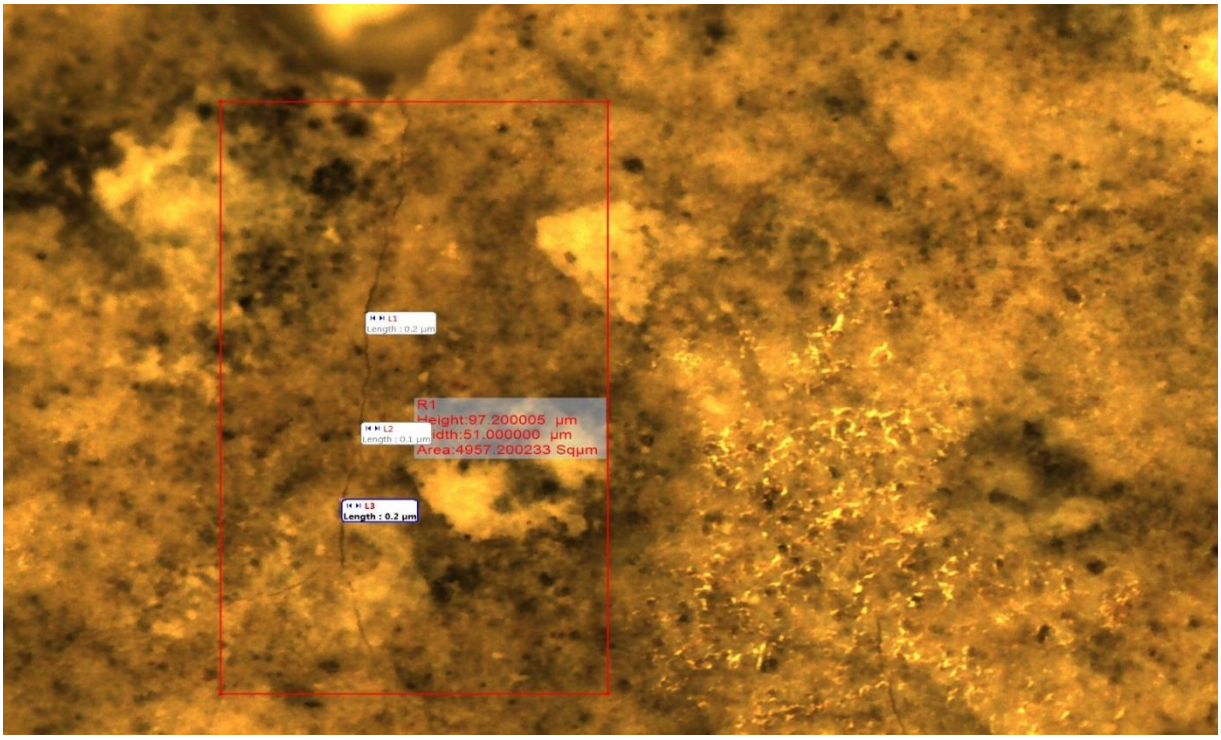


Figure 4.44 Cracks in 0.45 water to cement ratio

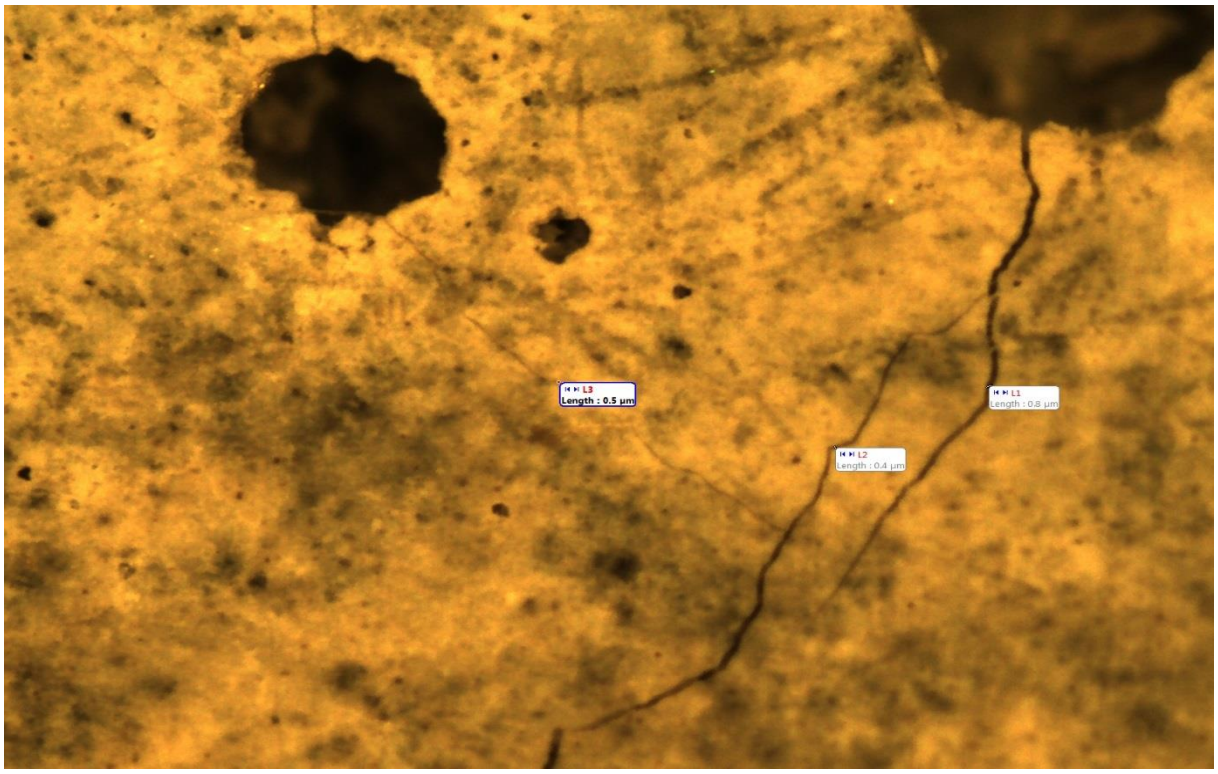


Figure 4.45 Cracks in 0.47 water to cement ratio

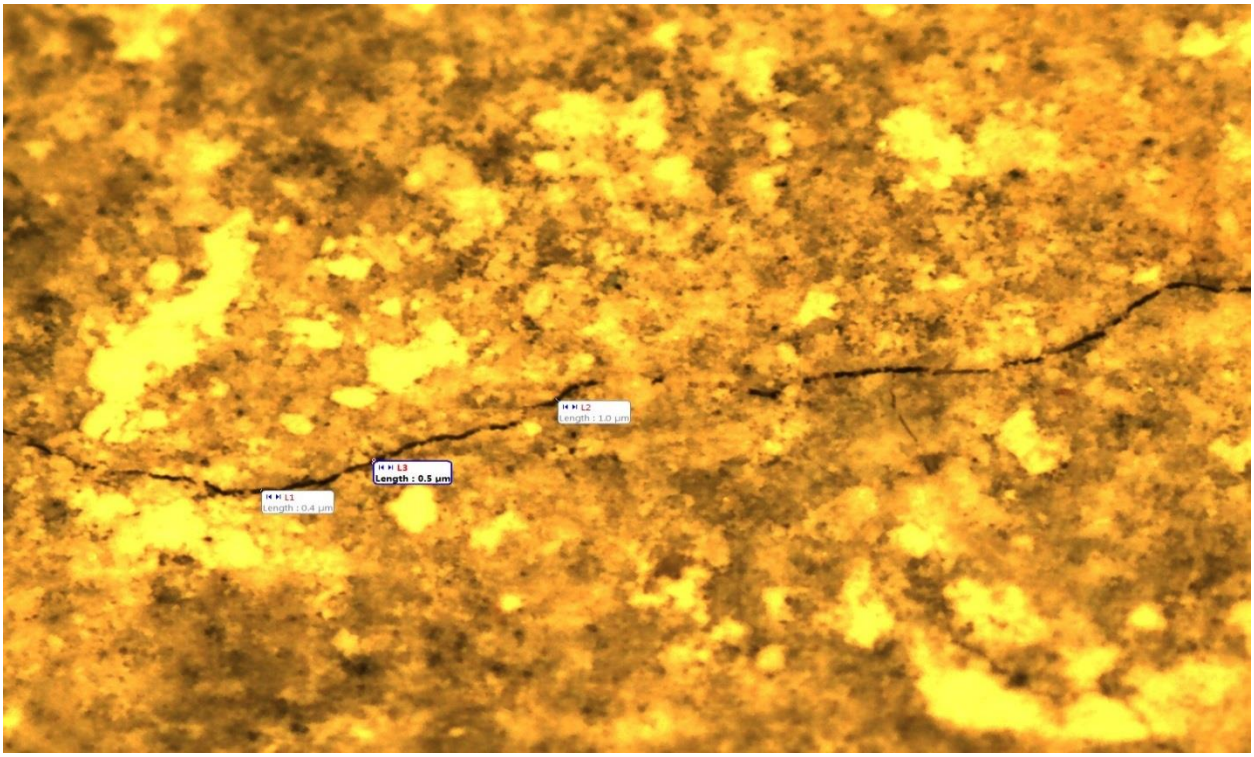


Figure 4.46 Cracks in 0.50 water to cement ratio

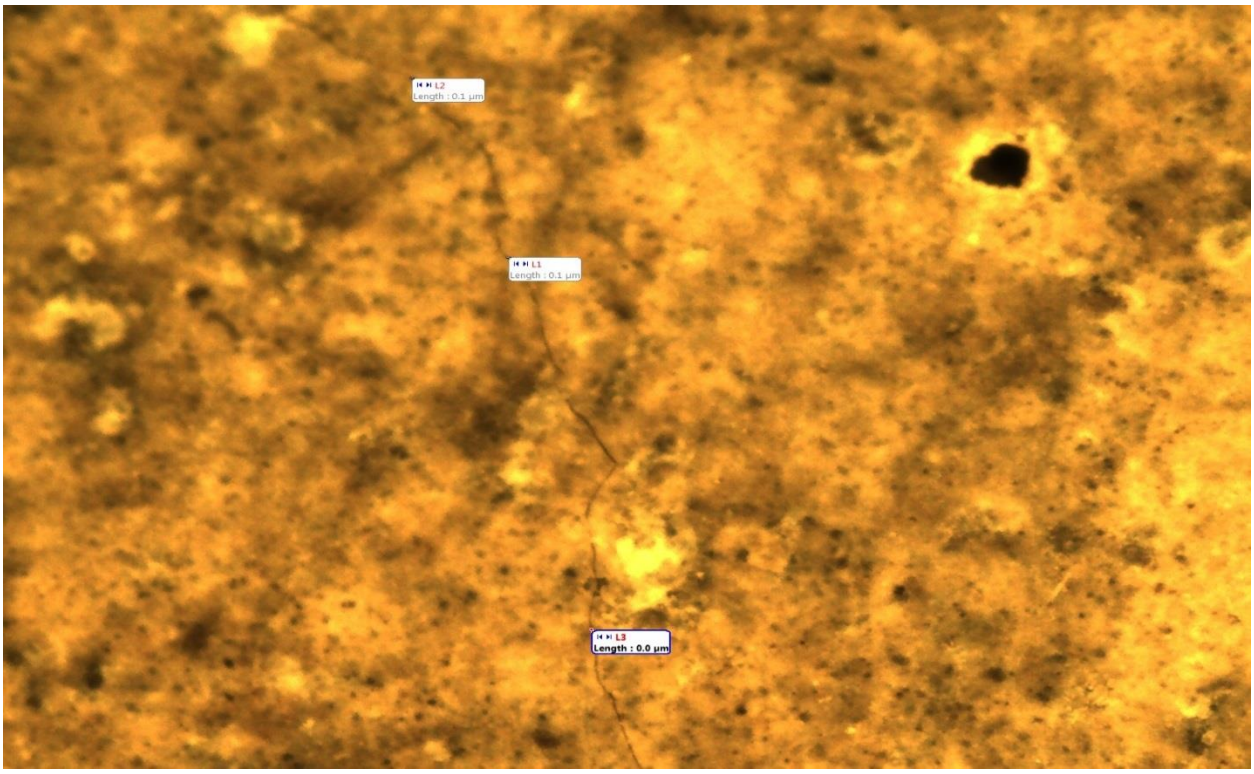


Figure 4.47 Cracks in 0.55 water to cement ratio

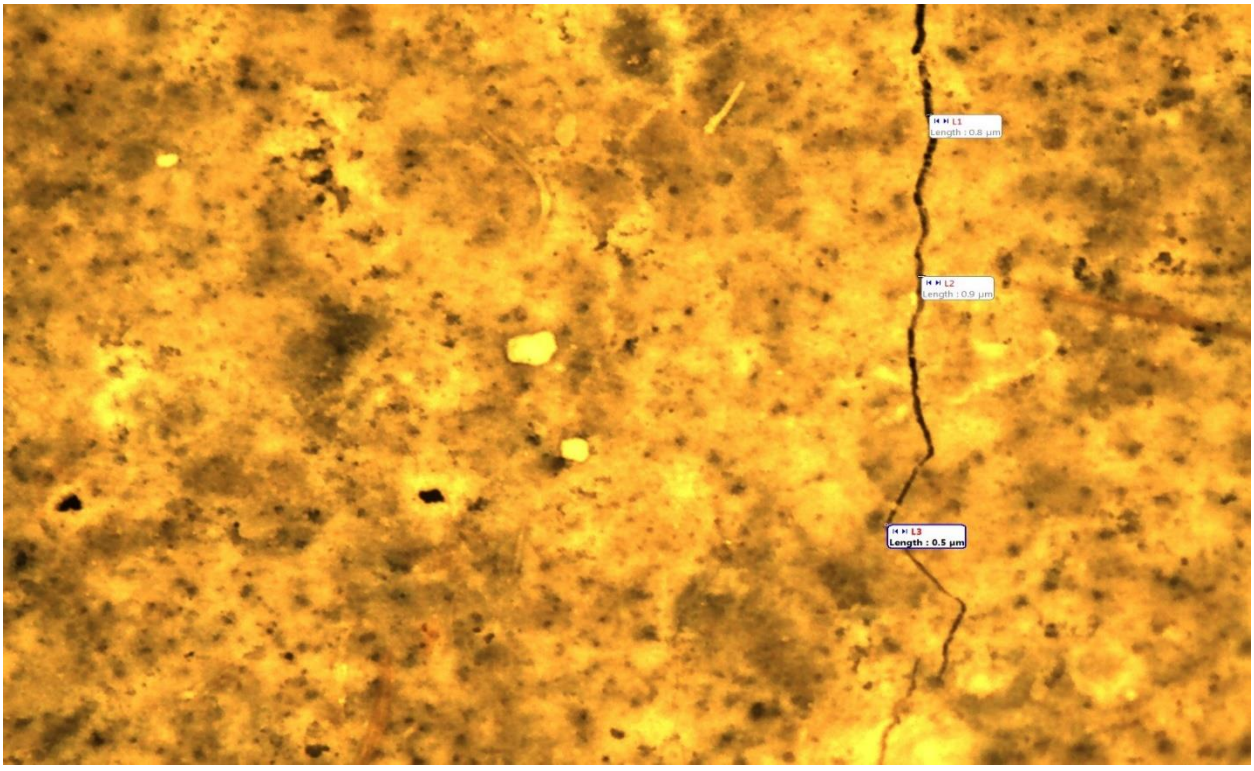


Figure 4.48 Cracks in 0.60 water to cement ratio

The width of the cracks was also measured at several points for each group of samples and the average is calculated for each water to cement ratio samples as shown in Table 4.1. It was interesting to find out that the average width of the cracks measured increase with the used water to cement ratio used to prepare the mortar samples. For the first three w/c (0.45, 0.47, 0.50), it was observed that the difference between the average width of crack measured for these water to cement ratios was higher compared to 0.55 and 0.60 water to cement ratio in which the difference in average width of cracks between these two water to cement ratio was lower.

Water to cement ratio	**Average width of cracks**
0.45	0.1667µm
0.47	0.5667µm
0.50	1.1667µm
0.55	1.6333µm
0.60	1.8667µm

Table 4.2. Average crack width for the five water to cement ratios

In order to assess the correlation between the mass of samples and progression of ASR, the mass of the samples was also measured alongside the surface area expansion and the results for all the water to cement ratio is presented in figures 4.49 to 4.53.

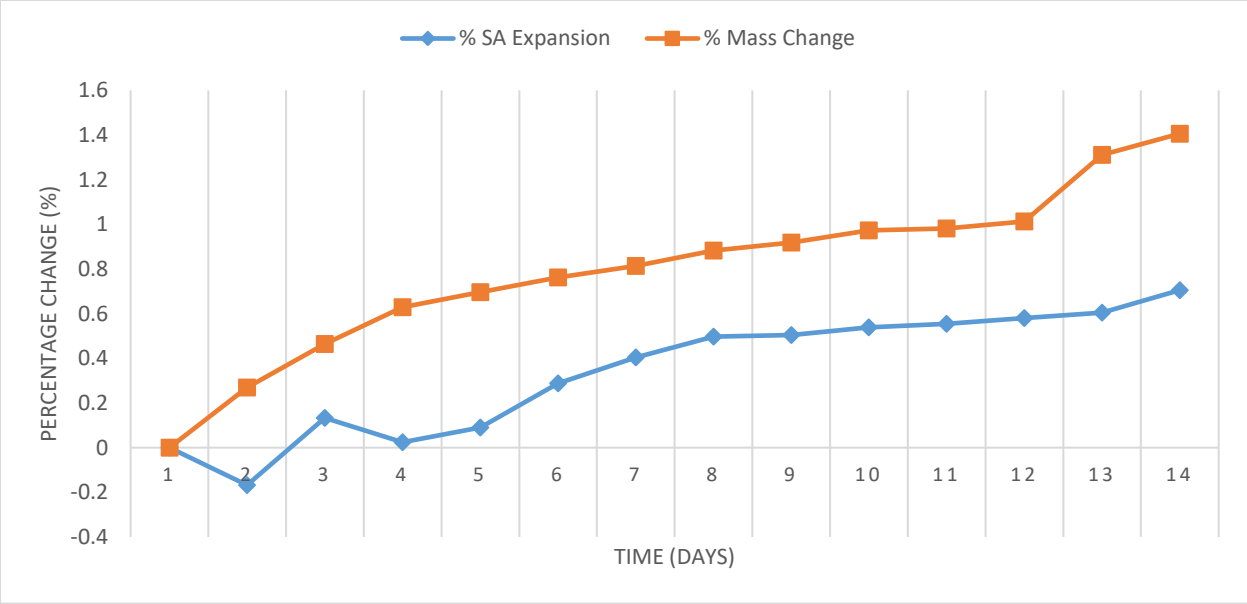


Figure 4.49 Correlation between percentage surface area expansion and percentage mass change for W/C 0.45

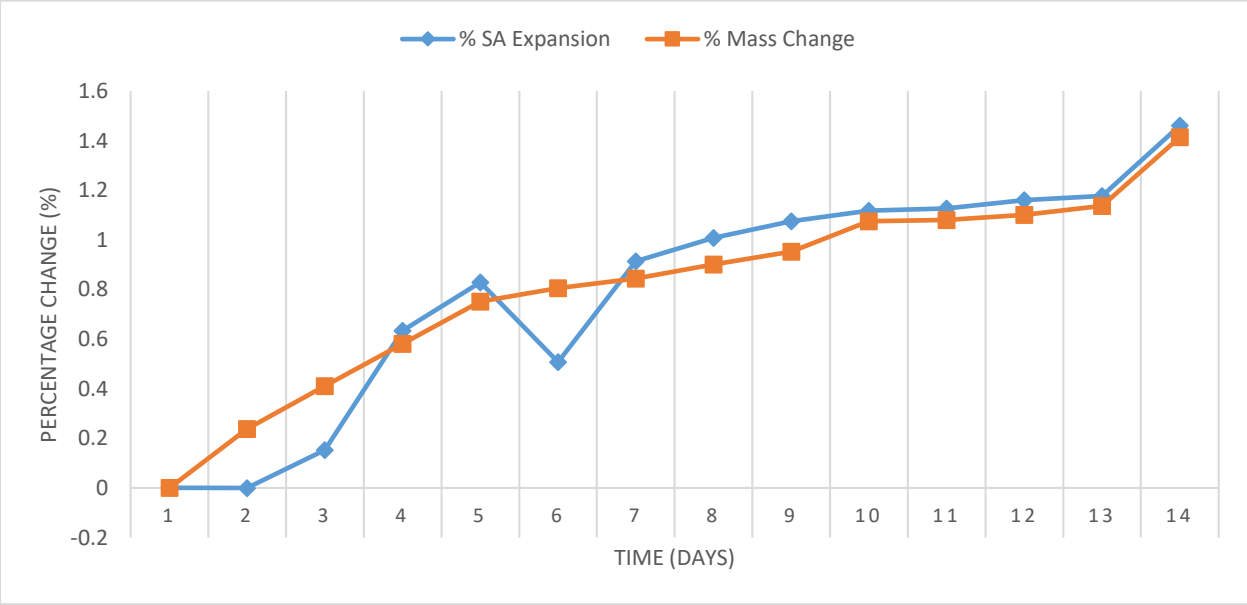


Figure 4.50 Correlation between percentage surface area expansion and percentage mass change for W/C 0.47

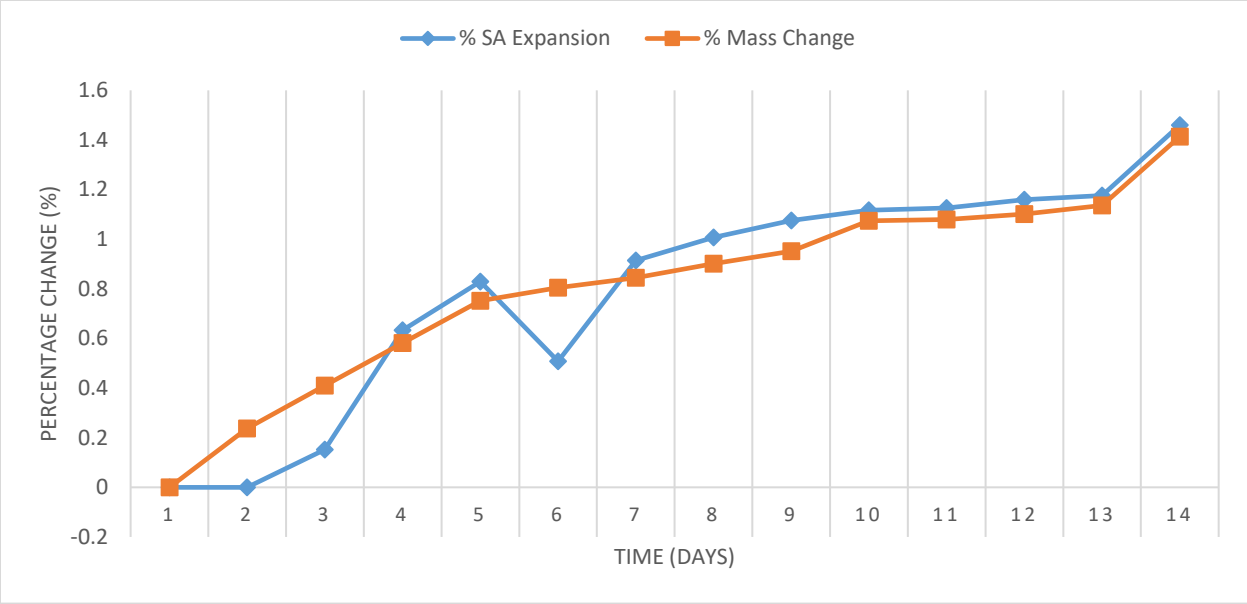


Figure 4.51 Correlation between percentage surface area expansion and percentage mass change for W/C 0.50

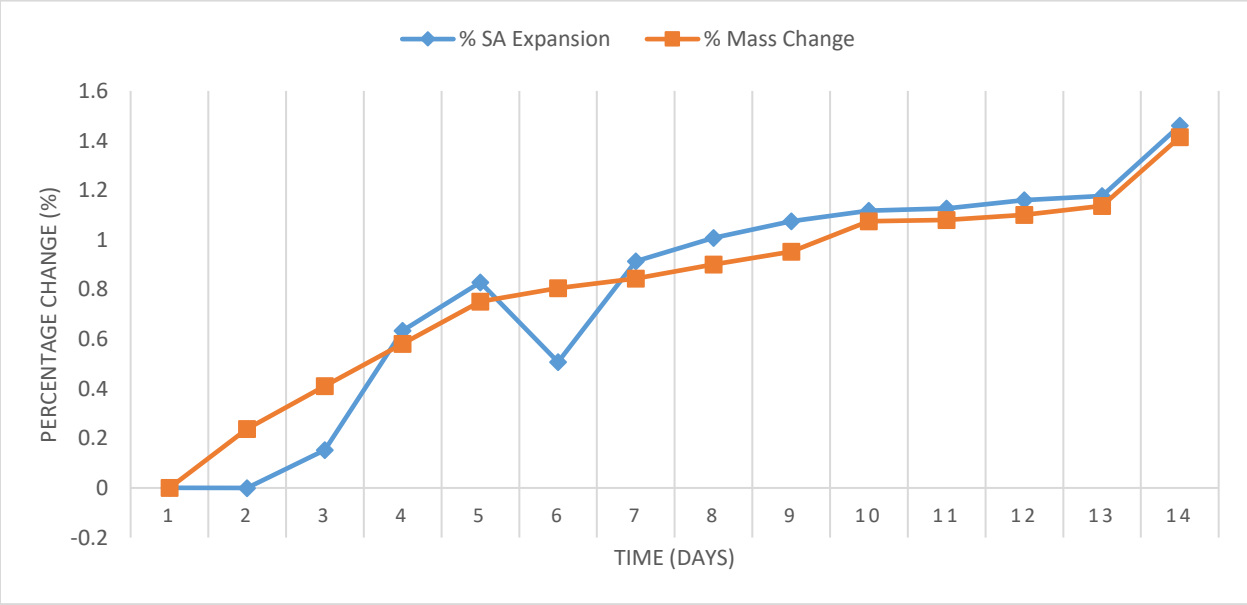


Figure 4.52 Correlation between percentage surface area expansion and percentage mass change for W/C 0.55

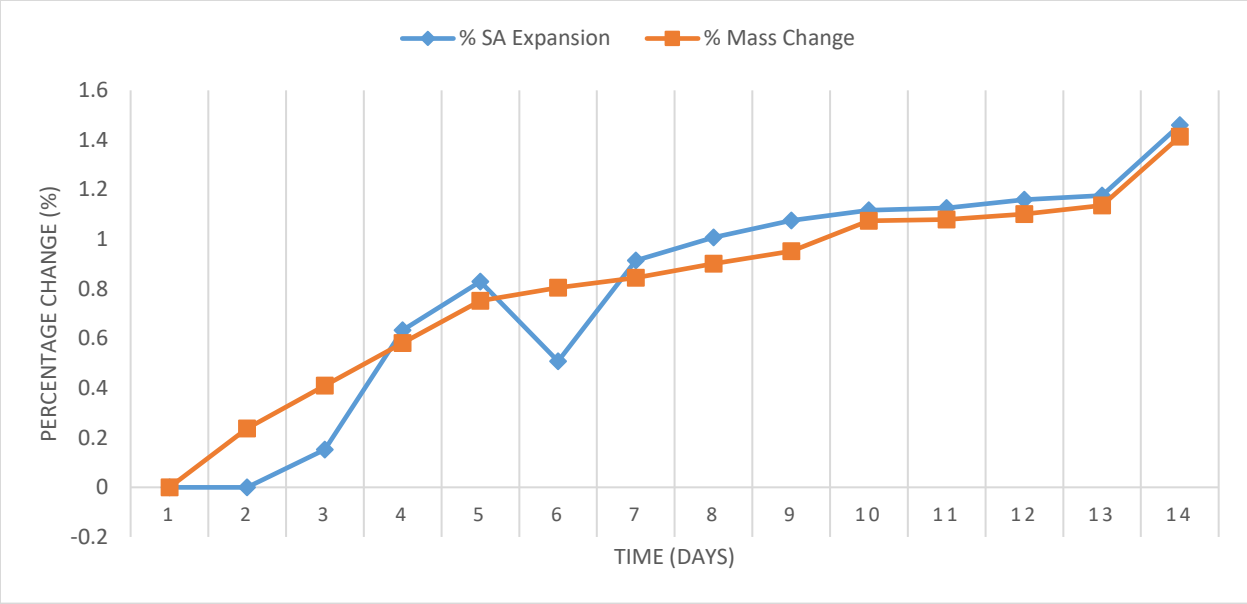


Figure 4.53 Correlation between percentage surface area expansion and percentage mass change for W/C 0.60

It can be observed from figures 4.49 to 4.53 for all water to cement ratio that there was an increase in the mass of the samples as the surface area increases, but there was no direct correlation in the rate of expansion of the sample and rate of increase in mass. The increase in mass of the sample might be as a result of the formation of silica gel and its expansion in the samples as it can be seen that there was increase in mass of all the samples throughout the 14 days.

From figure 4.43, it can be observed that the initial water to cement ratio has an effect on the expansion due to ASR. At 14 days, the percentage surface area expansion for w/c 0.45 and 0.50 is very low and close compared to the 0.47 water to cement ratio.

In samples with water to cement ratio 0.60, it was observed that the sample shrink more in the first day and then started expanding after that, and at 14 days, this mixture has the highest surface area

expansion compared to other water to cement ratios. This would be a result of the silica gel formed, being able to expand easily in the sample, since the tensile strength of the sample will be lower as compared to lower water to cement ratio, making it expanding without much restraint.

Based on the surface area expansions recorded, a visual survey of the sample was made using a microscope to observe if there are presence of surface micro cracks and cracks on the samples. Sample 2 (i.e. A2, B2, C2, D2, E2) of each water to cement ratio was observed and shown in figures 4.44 to 4.48. On all the samples observed, there were presence of cracks which vary in length and width, the average of the width for each water to cement ratio was presented in Table 4.1. Average width of cracks calculated for each water to cement ratio shows that the average width of the cracks of water to cement ratio of 0.60 was higher compared to others, and water to cement ratios 0.50, 0.55 and 0.60 have longer cracks compare to lower water to cement ratios and several cracks on the surface. The 0.47 and 0.45 water to cement ratio with fewer cracks and shorter length compared to others.

As it was observed based on previous results that the higher the water the cement ratio, the higher the expansion, it was also shown in the results in figure 4.49 to figure 4.53 that the higher the water to cement ratio, the higher the increase in mass of the sample in relation to the initial expansion. From the results obtained for all water to cement ratios for correlation between rate of surface area expansion and rate of change of mass, it was observed that though there is an increase in the masses of all the samples over time, but there is not direct correlation between the two criteria i.e. surface area expansion and mass change.

The water to cement ratio definitely contributes in a way to the development of expansion in ASR affected structure, though the effect might be minimal in big structures. Also, the development of ASR is related to the increase in mass of the sample as a result of the swelling of the ASR product formed which is the silica gel but there is not relationship between the ‘rate’ of expansion and ‘rate’ of mass gain.

5 CONCLUSIONS

5.1 CONCLUSIONS

With the overall objective of this thesis being to determine the effect of moisture on alkali silica reaction, the majority of the work done in this thesis focused on measuring relative humidity inside concrete. Relative humidity was used to quantify the amount of moisture in the experiments carried out and the experiments are divided into two phases. In phase one, an ideal sensor (iButton) was determined and used to measure the internal relative humidity at different locations in concrete samples so as to understand how the external relative humidity affects the internal relative humidity in concrete. In phase two, mortar cubes were made using Spratt reactive aggregate to observe the effect of external relative humidity on progression of ASR and effect of water to cement ration on ASR expansions.

Based on literature review, the relative humidity measured at a particular point in similar concrete samples should be in the range of ±2% and relative humidity in concrete was found out to be affected by the components present in concrete's composition and on it's surface. The effect of 50% fly ash replacement of cement and epoxy coating was investigated to see how these components affect the internal relative humidity in concrete compared to concrete samples composed of basic components (cement, aggregate and water). Also, all the samples were coated at the base and at the top to allow moisture loss and gain from only the surface except "coating" group of samples that are coated on all sides.

As a threshold of 80% relative humidity has been generally accepted for alkali silica reaction to occur, mortar cube samples are made to observe the expansions of the samples at different relative humidity but a fixed temperature of 38˚C as it was also suggested that expansions are possible below 80% relative humidity depending on the temperature (Olafsson 1986). Also, the initial water to cement ratio was examined as it is a source of water that cannot be eliminated but can be reduced. The mortar cubes were made using the ASTM C 1260 procedures with modifications on the mold for all experiments in phase 2 and modification of the water to cement ratio in phase 2b experiment. Conclusions made from all the experiments are summarized as follows:

Investigation of Sensors and Relative Humidity Measurement in Concrete Samples

1. The iButton DS 1293 is an ideal sensor to measure the internal relative humidity in concrete due to its energy self-sufficiency, data collection and retrieval and also in respect to its small size which allows placement in even smaller holes or to even to be embedded in fresh concrete.

2. In this research, the depth of the sensor did not show a consistent influence on the measured humidity.

3. The average internal relative humidity in samples exposed to 38ºC and 100% relative humidity varied between 99% ±1%. This is the same condition as ASTM C 1293 standard test conditions indicating that the high internal relative humidity will help to propel the rate of reaction in the samples to tests for the susceptibility of the aggregates to alkali silica

reaction.

4. Supplementary materials have an effect on the internal relative humidity of concrete. This might be as result of SCMs having a more compact pore structure compared to normal concrete which made the internal relative humidity measurements in concrete with 50% fly ash have a uniform pattern of gain and loss of moisture while the normal samples are not in any particular pattern.

Determination of the Effect of External Relative Humidity and Water to Cement Ratio on ASR

1. Mortar cubes exposed to 75% at 38˚C expanded as a result of ASR. Although this has already been observed by Olafsson (1986), this research confirms the expansion at this particular threshold. It was also shown that expansion is possible at lower relative humidities at 38˚C as observed in the results, though further tests would still be needed to verify this fully.

2. Progression of alkali silica reaction results in gain in mass of the mortar samples which might be as a result of the silica gel formed from the reaction absorbing moisture from the environment and thereby expanding. This agrees with claims of Tomosawa et al (1989), Kurihara et al, (1989) and Larive et al, (2000), but there was no direct relationship between the gain in mass and expansions observed.

3. In all the cube samples soaked in NaOH solution, expansions are recorded on all sides which might make the use of mortar cubes more accurate to test for expansions if an equipment that can be used to take precise measurements of expansion both axially and laterally compared to the current standards that measures only expansions axially.

5.2 RECOMMENDATIONS

1. The internal condition in concrete cannot be determined by just making reference to a point in concrete due to the complex concrete microstructure; therefore, several reference points have to be selected so as to get a true representation of the internal condition as concrete is a very complex material.

2. The use of Gore-Tex to cover the sensors so as to prevent it from the harsh alkali environment of concrete may have increased the response time of the sensors to humidity changes. It is suggested to further evaluation this effect.

3. In order to determine expansion of new aggregates to be used for construction, the lateral expansion (width) should also be measured and calculated not only the length change as there might be expansions laterally that won't be observed by just measuring the length changes as this was observed for experiment in phase 2 that expansions were recorded on all sides of the sample.

4. When measuring relative humidity in concrete, the sensors should be placed very close to the point being measured as a larger volume separating the point from the sensor can affect the measurement. In this research, the iButton sensor was placed at the bottom of the rubber stopper which gave a larger volume of air to achieve moisture equilibrium with the interior of the sample.

5.3 FUTURE STUDIES

As all the experiments done in the lab and with limited changes in environmental conditions, the following future studies will help to understand how moisture affects the progress of real structures in the field.

1. Embedding sensors in structures outdoors to measure the internal relative humidity in relationship with the external environment.
2. Measuring internal conditions in several outdoor structures affected by alkali silica reaction ranging from slight to severe.
3. Measuring the internal condition in various structures being mitigated against alkali silica reactions so as to know how effective the procedures and using a non-treated alkali silica reaction structure as control.
4. Determining a correlation between relative humidity and other forms of moisture measurement in concrete.
5. Testing more samples in the lab with various shapes and dimension to see the effect shape and dimension has on internal relative humidity in concrete.
6. Based on literature review, it has been shown that the temperature has huge effect on the relative humidity; this should also be studied in more detail.
7. As internal relative humidity is a huge concern that needs further research, developing appropriate procedure or standard to carry out the measurement will help to have a more accurate and reliable results.

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APPENDIX A

The measured relative humidity for each normal sample at each point are shown in this section

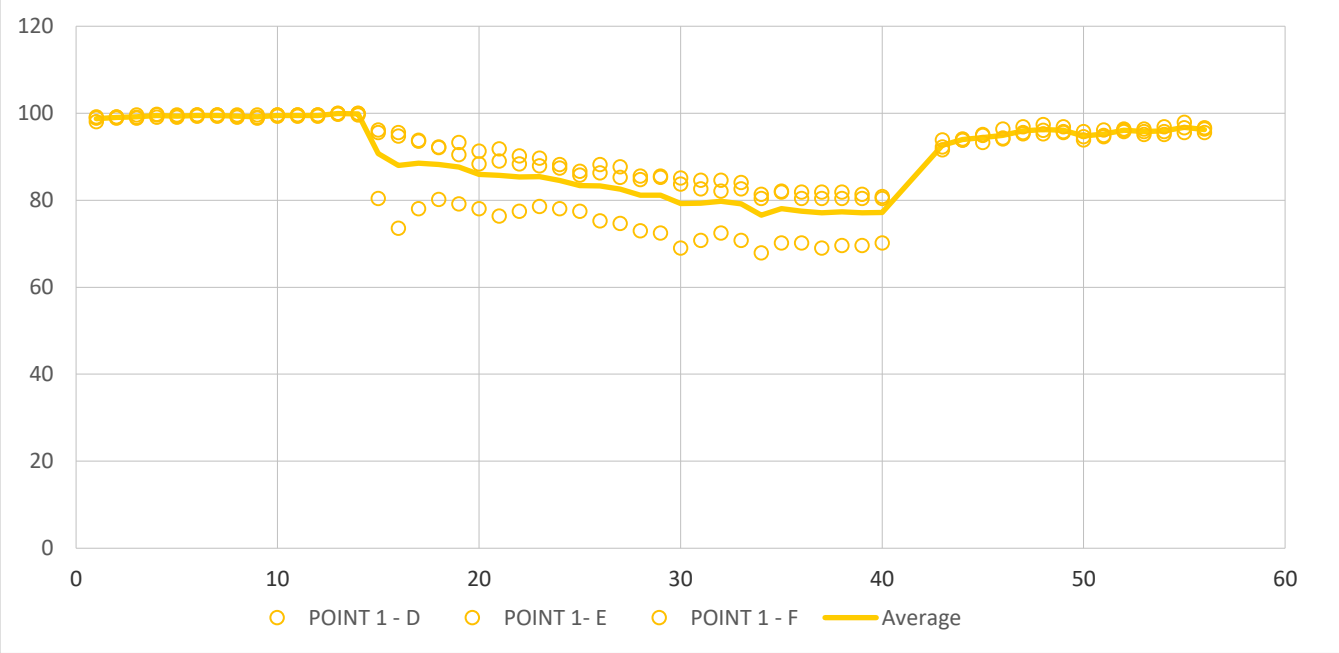

Figure A1. Measured Relative Humidity at Point 1 for normal samples

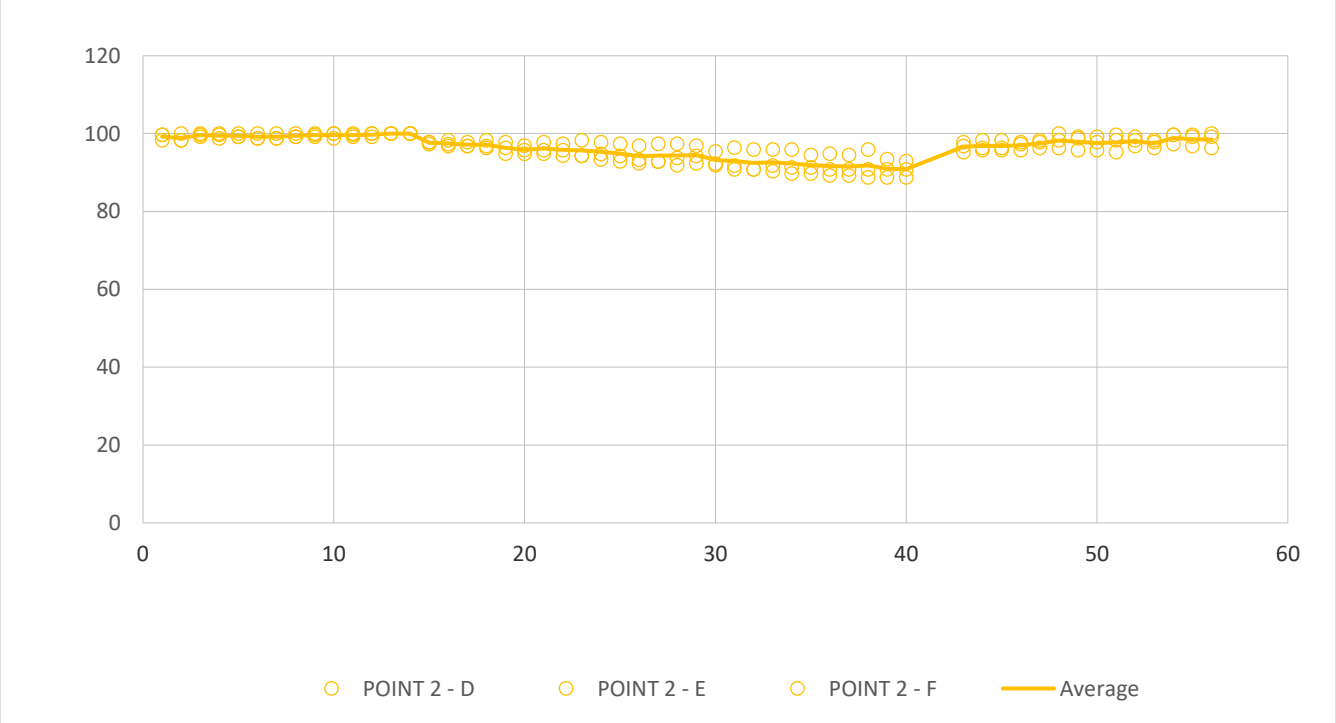

Figure A2. Measured Relative Humidity at Point 2 for normal samples

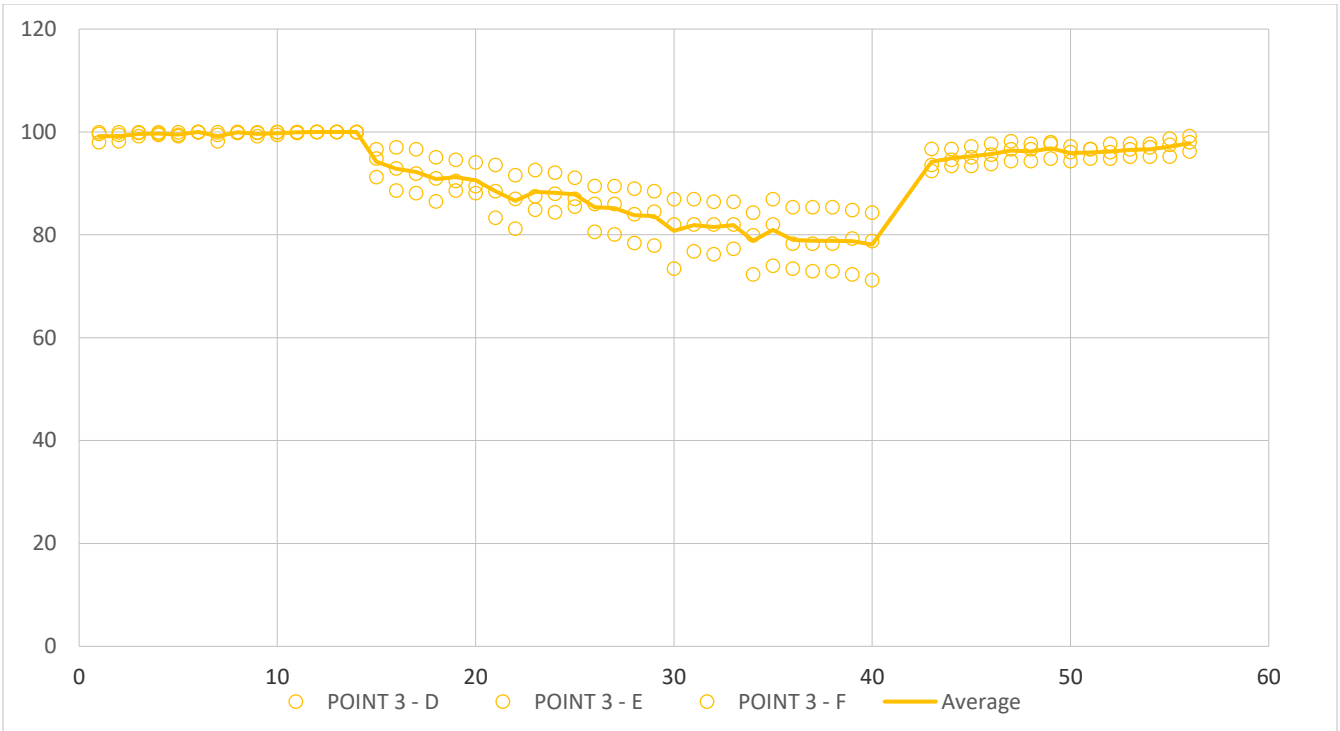

Figure A3. Measured Relative Humidity at Point 3 for normal samples